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## APPARATUS FOR MAKING X-RAY POWDER PHOTOGRAPHS AT CONTROLLED, ELEVATED TEMPERATURES

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### ABSTRACT

An x-ray powder camera for the investigation of substances at controlled, elevated temperatures is described. The apparatus differs in two respects from cameras previously described. In the first place, the film holder is removable without in any way disturbing the specimen or its condition in the furnace. In this way, the condition of the specimen may be independently controlled as desired while an exposed film is removed from the camera, developed, and another film substituted. This makes the apparatus especially suitable for the investigation of phase diagrams, for the specimen is never quenched or otherwise heat treated, except as desired. The apparatus also differs as to the method of maintaining the temperature of the specimen above room temperature, in that it is insulated against radiation losses rather than against conduction losses. This permits the construction of a furnace of negligible heat capacity, and because of this characteristic, the temperature of the specimen may be very rapidly varied, another feature of importance in phase diagram study. The temperature of the furnace at the specimen is calibrated by the investigation of substances having polymorphic transformations. The apparatus has been successfully employed in investigations up to about 600° C., and is suited to higher temperatures. The heating unit has also been adapted to the Weissenberg camera, which has been successfully used on a number of investigations of structural characteristics of single crystals at elevated temperatures.

### INTRODUCTION

A number of devices have been described for making x-ray powder photographs of materials held at elevated temperatures.<sup>1-12</sup> Most of these

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<sup>1</sup> Westgren, Arne, Roentgen spectrographic investigations of iron and steel: *Jour. Iron and Steel Inst.*, **103**, especially 306-309 (1921).

<sup>2</sup> Westgren, Arne, and Lindh, Axel E., Zum Kristallbau des Eisens und Stahls: *Zeits. phys. Chem.*, **98**, especially 192-194 (1921).

<sup>3</sup> Becker, Karl, Eine röntgenographische Methode zur Bestimmung des Wärmeausdehnungskoeffizienten bei hohen Temperaturen: *Zeits. Physik.*, **40**, 37-41 (1927).

<sup>4</sup> Westgren, Arne, and Phragmen, Gösta, Zum Kristallbau des Eisens und Stahls II: *Zeits. phys. Chem.*, **102**, especially 3-4 (1922).

<sup>5</sup> Cohn, Willi M., Über einem Röntgenofen zur Vornahme von röntgenographischen Untersuchungen bei hohen Temperaturen und über einige vorläufige Ergebnisse für Pentaerythrit und Quarz: *Zeits. Phys.*, **50**, 123-136 (1928).

are cumbersome and also inconvenient to use and are evidently intended to be used where the experimental part of the research requires making a single photograph. When phase diagram studies are to be made, it is either too tedious to make use of such instruments or else their characteristics actually prohibit their use.

As an outcome of experience gained in investigating the phase diagram of the system  $\text{Cu}_2\text{S}-\text{CuS}$  by making  $x$ -ray photographs of samples held at controlled, elevated temperatures,<sup>13</sup> we have designed an improved apparatus for this purpose, and it has now been successfully used over a period of about three years. This apparatus has the following important characteristics for phase diagram studies: The specimen is not in any way disturbed in its heat treatment when the film is removed for development. When a new film is returned, everything is in adjustment for the next photograph, to be taken, presumably, at a different temperature. A novel furnace design permits changing the temperature level of the specimen almost instantaneously. There are therefore no delays in waiting for the furnace to come to the desired temperature.

#### DESCRIPTION OF THE APPARATUS

*General Features.* Figure 1 shows a general view of the assembled apparatus. It fits, by means of a dovetail slide, onto a bracket which also supports the  $x$ -ray tube, Fig. 2. This arrangement permits the camera assembly to be easily removed and returned to its position with respect to the  $x$ -ray tube. The sliding motion of the camera is limited by an adjustable stop which permits recovery of exact position. The camera is adjusted to the  $x$ -ray beam as follows:

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<sup>6</sup> Jay, A. H., A high temperature  $x$ -ray camera for quantitative measurements: *Zeits. Krist.*, (A) **86**, 106-111 (1933).

<sup>7</sup> Wilm, D., Hofmann, U., and Endell, K., Über die Bedeutung von Röntgeninterferenzuntersuchungen bei hohen Temperaturen für die keramische Forschung: *Sprechsaal für Keramik, Glas, Email, Fach- und Wirtschaftsblatt*, Nr. **38**, 1-8 (1934).

<sup>8</sup> Finbak, Chr., and Hassel O., Kristallchemie der Nitrate einwertiger Kationen, II: *Zeit. Chemie* (B) **37**, 75-80 (1937).

<sup>9</sup> Schossberger, F., Eine Präzisions-Pulverkamera für Aufnahmen bei hohen Temperaturen und ein Messgerät für Röntgendiagramme: *Zeits. Krist.*, (A) **98**, 259-265 (1937).

<sup>10</sup> Straumanis, M., and Ieviš, A., Die Bestimmung von Ausdehnungskoeffizienten nach der Pulver- und der Drehkristal-Methode: *Zeits. anorg. allg. chem.*, **238**, especially 180-181 (1938).

<sup>11</sup> Straumanis, M., Die Gitterkonstanten und Ausdehnungskoeffizienten des Tellurs und Selens: *Zeits. Krist.*, (A) **102**, 432-454 (1940).

<sup>12</sup> Wilson, A. J. C., The thermal expansion of aluminium from 0° to 650° C.: *Proc. Roy. Soc. Lond.*, **53**, 235-244 (1941).

<sup>13</sup> Buerger, Newton W., The chalcocite problem: *Ec. Geol.*, **36**, 19-44 (1941).

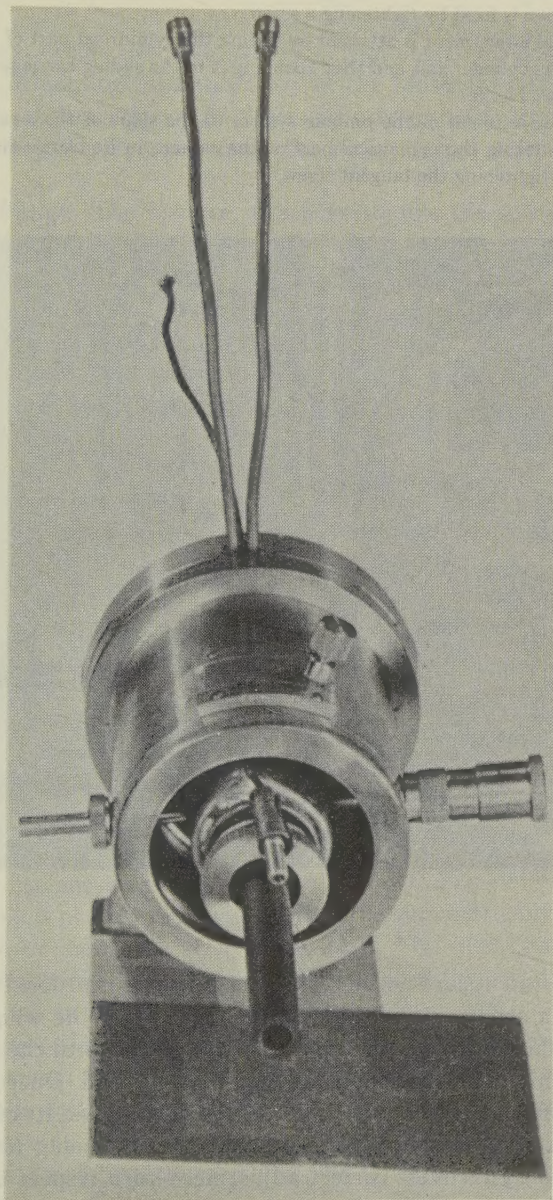


FIG. 1

(1) Vertical adjustment is achieved by sliding the bracket up and down on a keyed slide. Final position is fixed by tightening a screw.

(2) Horizontal adjustment is attained by sliding the cylindrical part of the camera in the direction of its cylinder axis and then clamping it to the sliding carriage by tightening the tangent screw, Fig. 6.

(3) Angular adjustment of the pinhole system to the slope of the  $x$ -ray beam is accomplished by rotating the cylindrical part of the camera in its clamp on the carriage, Fig. 6, and then tightening the tangent screw.

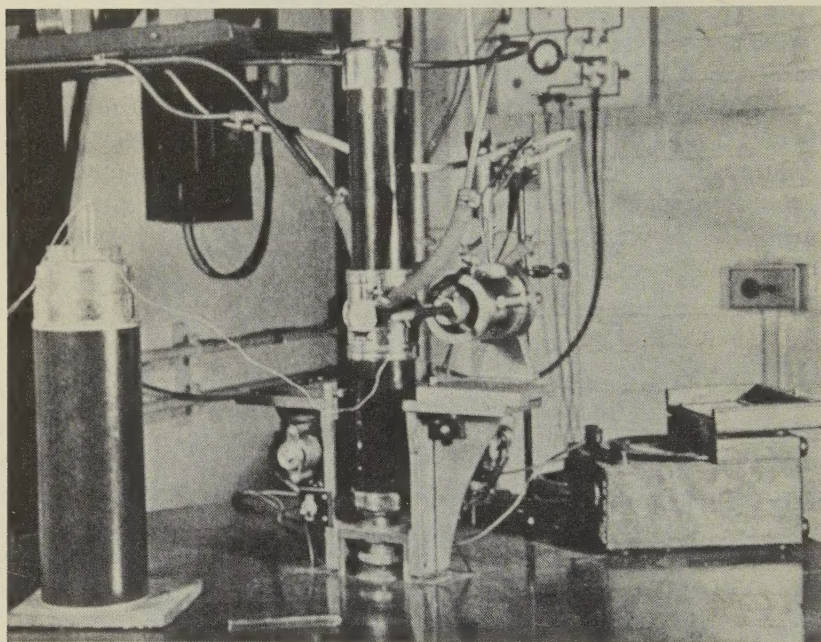


FIG. 2

These adjustments are not necessarily made in this order. It is possible to loosen both screws mentioned above, and adjust the whole bracket-camera assembly in the manner of a universal joint, until the  $x$ -ray beam is seen emerging in the exit port, as described beyond. Once the adjustments mentioned above have been made, the camera and its carriage may be removed at any time from the bracket and, on being returned, the apparatus exactly recovers correct adjustment with respect to the  $x$ -ray beam.

The bracket also carries a small Telechron motor which rotates the specimen. The motor is mounted on an insulating support and drives the specimen shaft through a rubber band belt. This precaution prevents

burnouts of the motor at times of temporarily erratic operation of the x-ray tube.

The general design described above is common to all powder cameras used in the Mineralogical Laboratory of the Massachusetts Institute of Technology, and in respect to these features and those of the camera proper (see beyond) they supersede the earlier design described by M. J. Buerger.<sup>14</sup>

*Furnace Design.* The furnace, which surrounds the specimen, is a removable unit, and is shown slipped off from its position at the left of Fig. 5*B*. In Fig. 5*C* it is shown in position.

The furnace unit is illustrated in detail in Fig. 4. It is believed that the system for maintaining temperature herein described is unique in furnace design. It is customary to maintain temperatures by surrounding the heating unit with insulation, i.e., by damming the heat flow by conduction barriers. In the tiny furnace here described, the temperature is maintained by damming the heat flow by radiation barriers. This is done by placing two very thin hollow aluminum cylinders about the heating unit. This device is nearly opaque to heat radiation but is transparent to the x-radiation diffracted by the specimen. This kind of heat barrier has the advantage over conduction insulation in that it occupies almost no space, since its functional barriers are surfaces, while the efficiency of conduction insulation depends upon its thickness, which causes it to occupy appreciable volume. Radiation barriers have the further advantage that they have negligible heat capacity, and so the temperature of the furnace can be very rapidly, almost instantaneously, changed to a new level at pleasure.

The actual furnace takes the form of a small capsule shown assembled in Fig. 3(*e*). This contains several concentric elements. The inner one is the heating element, a coil of #28 B. and S. gauge chromel "A" resistance wire. This coil is prepared by winding 9 turns symmetrically on each side of a  $\frac{3}{16}$  inch diameter mandrel as shown in Fig. 3(*a*). This method of winding deliberately leaves a gap in the center of the coil through which the direct x-ray beam is to pass.

The heating coil is mounted by allowing it to expand within a porcelain tube (dimensions: 1 inch long and  $\frac{3}{8}$  inch O. D.) whose chief function is to support the coil. To permit the diffracted beam to emerge from this tube, a transverse slot is cut in the porcelain, Fig. 3(*b*), covering all but about 70° of its circumference. This slot is widened somewhat at two diametrically opposite points to permit the direct x-ray beam to enter and leave without striking the porcelain.

<sup>14</sup> Buerger, M. J., An x-ray powder camera: *Am. Mineral.*, **21**, 11-17 (1936).

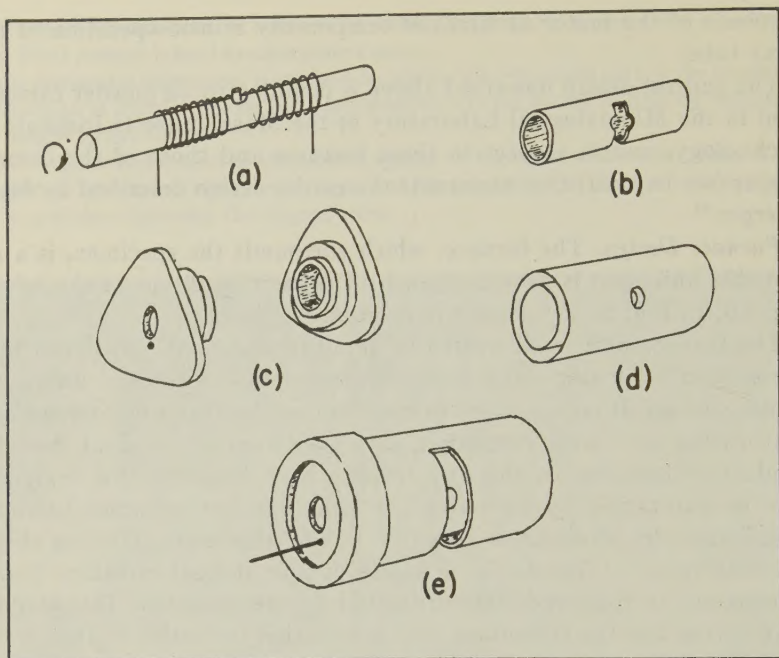


FIG. 3

The porcelain tube with its contained coil is thrust into two Transite spool ends, Fig. 3(c). These serve to axially centralize the tube in its protective copper cartridge, Fig. 3(e). The spool ends are not quite circular, but so shaped that they are supported within the cartridge on three circumferential points, thus minimizing conduction and consequent heat loss to the cooled copper.

The spool ends also support the aluminum foil radiation barriers. To this end they have two circular steps, Fig. 3(c); each supports a hollow cylinder of very thin aluminum foil (.0004 inch thick). Each aluminum cylinder is pierced with two diametrically opposite holes, Fig. 3(d), coinciding in position with the widened points of the slot in the porcelain cylinder. In assembling the furnace unit, all holes must be carefully lined up to permit the direct *x*-ray beam to enter and leave the cartridge. The technique of forming the aluminum cylinder is as follows. The sheet of foil, with entrance and exit holes carefully placed in computed positions, is laid on a flat piece of metal of its own width, and carefully shellacked on the edges where the foil is to stick to the spool and where it is to overlap itself. The spool is then rolled over the flat foil in the direction of its length, thus causing the foil to stick itself to the circular shelves on the spool ends and form the hollow cylinder.

The heating unit is placed within a copper cartridge, Fig. 3(e), for protection in handling. This has a slot coinciding with that of the porcelain cylinder in order to pass diffracted radiation from the heated specimen. As indicated in Figs. 4 and 3(e), the copper cartridge has an open back and front to minimize contact with the spool ends and thus reduce heat loss by conduction of transite to cold copper at this point. The skeleton cover has a screw fit.

One side of the heating coil winding is grounded to the cartridge. The other end protrudes from the front, insulated by the transite, Fig. 3(e).

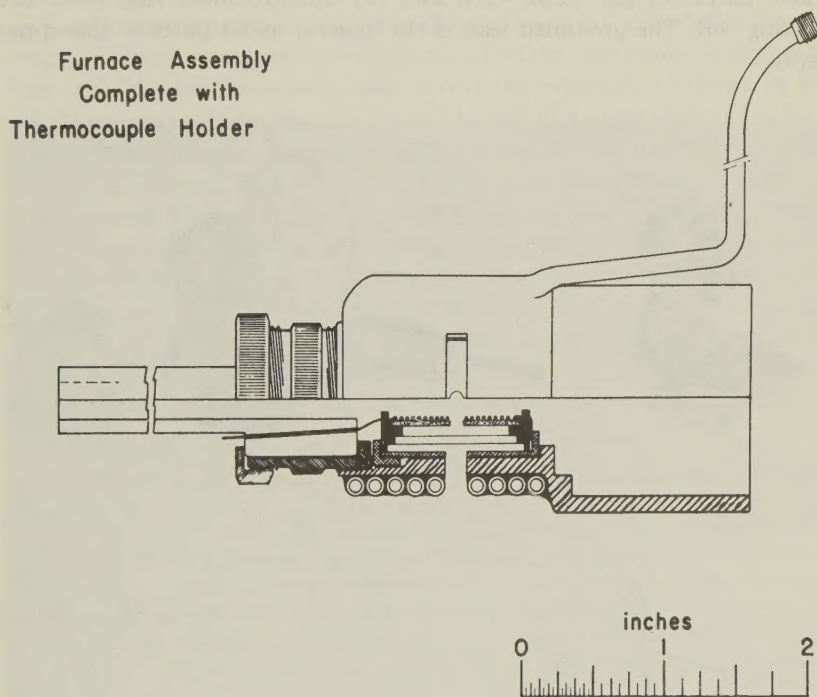


FIG. 4

The heating unit cartridge is held in place within a water-cooled jacket, Fig. 4, by means of a threaded metal member. The non-grounded lead to the heating coil is insulated from this by being led out through a fiber member, which also serves to hold a thermocouple, should this be desired (see beyond). The larger cylindrical jacket, Fig. 4, is wound with  $\frac{1}{8}$  inch copper tubing for water cooling in the region of the cartridge, with the usual opening provided to pass diffracted radiation. The tubing is made an integral part of the jacket by being wiped with solder until all space

between coils and jacket is filled and the whole mass of metal becomes a unit. This cooling jacket has two functions:

- (1) It protects the film against damage due to the high temperature of the furnace.
- (2) It provides the apparatus with a definite ground-level of temperature. A definite amount of electrical energy is required to raise the apparatus by a definite temperature interval,  $\Delta T$ , above this ground-level. Were it not for the cooling jacket, this ground-level would vary unpredictably with a number of external conditions.

The entire furnace jacket assembly, Fig. 5B, plugs into the camera spindle assembly as shown in Fig. 5C. From it projects three leads: (1) water entrance, (2) water exit, and (3) non-grounded lead from the heating coil. The grounded lead is the general metal parts of the apparatus.

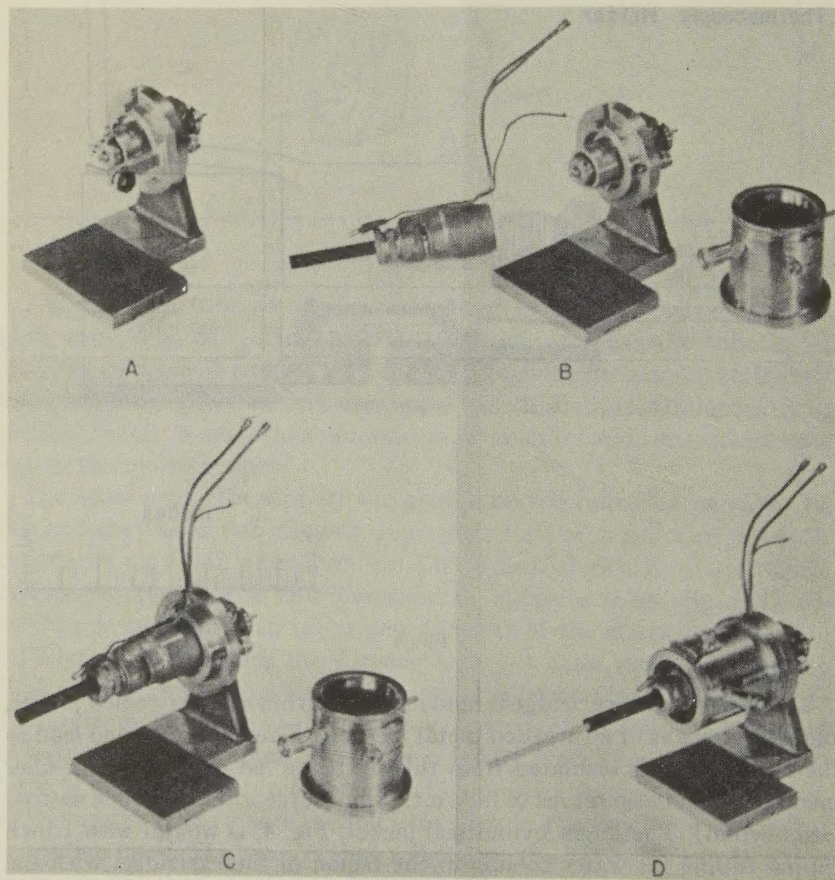


FIG. 5

*Specimen-Holding and Centering Device.* The specimen is prepared by the method of Buerger<sup>15</sup> or by the method described by Lukesh,<sup>16</sup> or, in certain instances, is sealed within a glass capillary. The metal specimen holder is placed in a centering chuck which differs from that used in the ordinary powder camera by having removable adjusting knobs, Fig. 5A.<sup>17</sup> These are really small screwdrivers (outer knobs) which are held in place on the chuck by taper plugs (inner knobs). These screw drivers are plugged in during centering operations and removed when this is completed so that the chuck and specimen can be retracted for reasons mentioned below.

A great convenience in centering the specimen is a detachable "microscope." This is arranged to fit during centering operations on the same base which holds the camera unit during the exposure, as shown in Fig. 5A. It consists principally of a lens at the end of a brass tube of fine bore. The lens is located at a distance from the specimen equal to its focal distance.<sup>18</sup> When looking at the specimen through this system, the eye sees the specimen framed in the aperture at the far end of the tube. Before adjustment, the specimen, when rotated, usually appears to move up and down in this aperture. By adjusting the milled knobs of the screwdrivers, the specimen is caused to become concentric with the axis of rotation, after which it appears to remain stationary in the aperture of the microscope when the shaft is rotated.

When the specimen adjustment has been accomplished, the following changes in the apparatus setup are made, in this order:

- (1) The microscope is removed by unscrewing the milled nut shown at the right of Fig. 5A.

- (2) The screwdriver adjusters for the specimen centering device are removed as follows: The outer, screwdriver handles are pulled outward to disengage them and thus avoid spoiling the adjustment. Then the taper plugs are removed by pulling on the inner knobs with a rotating motion. The apparatus then appears as in Fig. 5B.

- (3) The specimen is retracted into the hollow central hub of the camera base by pulling the shaft and pulley to the right. This protects the specimen while attaching the furnace unit.

- (4) The furnace, Fig. 5B, left, is slipped onto the central hub of the camera holder in such a way that the non-grounded wire and the cooling water leads fit into the recess in the camera base. The apparatus then appears as shown in Fig. 5C.

- (5) The shaft and pulley are pushed back so that the specimen enters the small axial hole in the furnace. A recoverable final position for the specimen is assured by the following arrangement: The specimen shaft has a small circumferential groove turned into it at a

<sup>15</sup> Buerger, *op. cit.*, p. 16.

<sup>16</sup> Lukesh, Joseph S., An improved technique for mounting powdered samples for x-ray diffraction: *Rev. Sci. Instruments*, **11**, 200-201 (1940).

<sup>17</sup> Buerger, *op. cit.*, p. 14.

<sup>18</sup> Buerger, M. J., The precision determination of the linear and angular lattice constants of single crystals: *Zeits. Krist.*, (A) **97**, 441 (1937).

convenient place which normally lies within the bearing. The shaft is normally retained in this place by a small ball which lies in the bottom of a transverse drillhole in the bearing. When the shaft is translated out of position as in (3) and then restored, the ball falls into the groove in the shaft when the shaft reaches the correct position and prevents the shaft from moving axially. The ball is maintained against the shaft by means of a weak spring

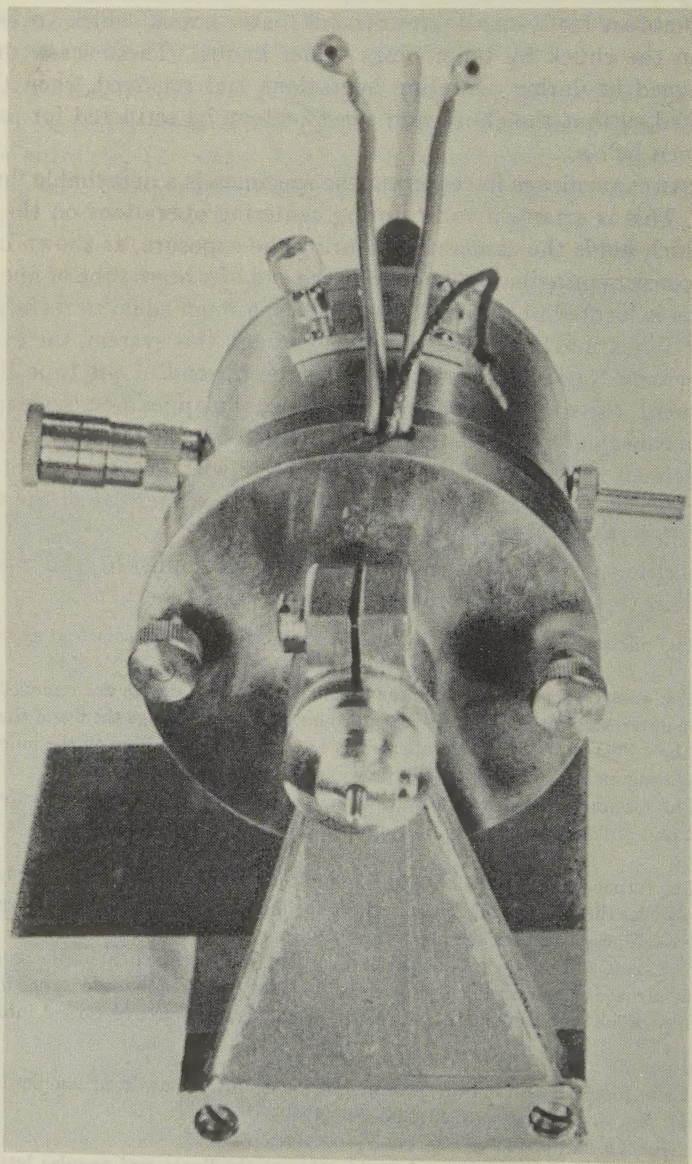


FIG. 6

(6) The loaded camera described below is slipped into a circular base, and fixed firmly in this position by tightening the milled knob shown in Fig. 6. The final appearance of the assembled apparatus is shown in Fig. 5D.

*The Camera Unit.* The camera proper has been deliberately designed so that it can be removed for development and replacement of the film while the specimen remains undisturbed in the furnace. This permits controlling the heat treatment of the specimen independently of making an x-ray photograph of its condition.



FIG. 7

The assembled camera unit for the 57.3 mm. diameter camera is shown at the right of Fig. 5B. Figure 7 shows this unit dismantled for the removal of the film. The film is placed in the camera Straumanis-fashion.<sup>19,20</sup> The reason for this is not so much to utilize Straumanis' strategy for determining the camera diameter, as to place the ends of the film in a position of a desirable blind spot (above the nonslotted part of the heating unit, Figs. 5C and 4). The furnace unit must have a blind spot, and this particular position permits having one continuous film record of reflections from  $2\theta = 0^\circ$  to  $2\theta = 180^\circ$ . This position also provides a doubled record in the back reflection field so that the Bradley and Jay<sup>21</sup> strategy for refining lattice constants can be used and at the same time shrinkage errors can be completely eliminated as described by Cohen.<sup>22,23,24</sup>

The film is spread against the inside of the brass camera drum by means of a pair of claws, one fixed and the other movable. The inside of this device is shown in Fig. 7A, and the outside in Fig. 5D. In loading, the film is placed somewhat loosely in the camera and then the spreader is operated and locked in the spread position. This automatically centers the film so that the holes for entrance and exit of the  $x$ -ray beam come accurately to position opposite corresponding holes in the camera cylinder. The film holes are neatly punched with a device which simultaneously cuts a 1×7 inch strip from a standard 5×7 inch double coated  $x$ -ray film.

After the film is in position, it is protected from light by slipping within the camera drum, another drum (left of Fig. 7A) which covers the film. The cover of this drum screws into the top of the film-holding drum and at the same time the bottom of the inside drum fits into a recess in the film-holding drum. The latter arrangement acts as a light-maze and prevents fogging of the film. The diffracted  $x$ -rays reach the film through a slit in the inner drum, Fig. 7B, and light is prevented from using the

<sup>19</sup> Straumanis, M., and Ievņš, A., Präzisionsbestimmung von Glanzwinkeln und Gitterkonstanten nach der Methode von Debye und Scherrer: *Naturwissenschaften*, **23**, 833 (1935).

<sup>20</sup> Straumanis, M., and Ievņš, A., Präzisionsaufnahmen nach dem Verfahren von Debye und Scherrer. II: *Zeit. Physik*, **98**, 461, 462, 466 (1936).

<sup>21</sup> Bradley, A. J., and Jay, A. H., A method of deducing accurate values of the lattice spacing from  $x$ -ray powder photographs taken by the Debye-Scherrer method: *Proc. Phys. Soc.*, **44**, 563-579 (1932).

<sup>22</sup> Cohen, M. U., Precision lattice constants from  $x$ -ray powder photographs: *Rev. Sci. Instruments*, **6**, 68-74 (1935); **7**, 155 (1936).

<sup>23</sup> Cohen, M. U., The elimination of systematic errors in powder photographs: *Zeits. Krist.*, (A) **94**, 288-298 (1936).

<sup>24</sup> Cohen, M. U., The calculation of precise lattice constants from  $x$ -ray powder photographs: *Zeits. Krist.*, (A) **94**, 306-310 (1936).

same path by covering the slit with black paper. The light-tightness of the film chamber is finally made complete by inserting the entrance port, (which is the pinhole system), and the exit port, Fig. 7*B*. These are both turnings with shelves in such positions as to form light-mazes with the drums.

The exit port has a number of functions. Besides permitting the beam to leave the camera, it envelopes the *x*-ray beam path on the exit side of the specimen in the camera and thus prevents *x*-rays scattered by the air along the beam path from reaching the film. The end of the port is fitted with a disk of fluorescent screen which permits the operator to check the adjustment of the pinhole system and specimen in the *x*-ray beam at any time. The operator is protected from the *x*-ray beam by a disk of lead glass at the end of the exit port.

The entire camera unit, Fig. 5*B*, right, fits against its circular base in such a way as to recover exact position each time. This is assured by accurate concentricity of the two turnings. The camera is held in place against the base by means of a couple of non-removable screws with milled handles, shown in Fig. 6. The assembled camera is shown in Figs. 1 and 5*D*.

*Operation of the Furnace.* The temperature of the furnace is regulated by connecting the heating coil in series with a slide wire rheostat for coarse adjustment and a carbon-block rheostat for fine adjustment. Satisfactory operation cannot be achieved by connecting this electrical sequence directly to a 110-volt power supply, for experience has shown that the ordinary power source is much too variable to maintain a constant temperature in the furnace. (A voltage stabilizer might be used to improve the voltage constancy of such a source.) We solved the problem of a constant source by utilizing storage batteries. With the apparatus described, one 6-volt storage battery is sufficient for operating at temperatures up to about 200° C. and two batteries in series are sufficient for temperatures up to about 600° C. For phase diagram work, we keep an equal number of batteries constantly charging, and change batteries every 24 hours of continuous run. When the change is made, care must be taken to watch the output drop in the battery during the first hour after charging. By the end of this time, its output approaches sufficient constancy until the battery approaches exhaustion, and the temperature of the furnace can be maintained constant to within about a degree.

*Calibration of Furnace Temperatures.* It might be supposed that the temperature of the furnace could be adequately determined with the aid of a thermocouple. Unfortunately this is not the case; a thermocouple always indicates too low a reading, as determined by the more accurate method described beyond. This is due to the fact that the tiny furnace does not

heat the entire thermocouple, but only the junction; consequently the thermocouple leads, which are colder than the junction, conduct this tiny amount of heat away rapidly, lowering the temperature of the thermocouple junction below that actually supplied by the furnace. The position of the thermocouple, if used, is shown in Fig. 5D.

Our method of calibration is to make use of polymorphism. If a crystalline substance which exhibits a polymorphic transition of the rapid variety is used as the specimen, the temperature is known at which its x-ray pattern changes. If the electrical energy supplied to the furnace windings to obtain two x-ray patterns, one on each side of the polymorphic change, is recorded, then some energy reading between these corresponds with the known temperature of the polymorphic change.

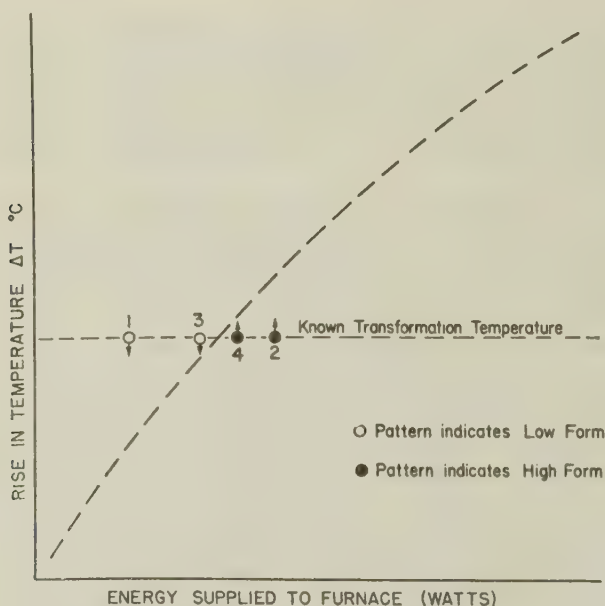


FIG. 8

Figure 8 illustrates the technique of determining the minimum energy supplied to the furnace sufficient to bring about the transformation. The trials are numbered 1, 2, 3, and 4. A photograph is first made with the specimen maintained at an energy setting 1; suppose that the pattern proves to be that of the low form. A higher energy setting 2, is then tried; the pattern then proves to be that of the high form. The true energy required for the transformation temperature, therefore, lies between readings 1 and 2. Adjustments are then made to give an energy reading,

3, halfway between readings 1 and 2. Suppose that the photograph proves that the crystal is now in the low form. The correct energy reading for the transformation then lies between readings 2 and 3. The rheostats are therefore set to give a reading 4, halfway between these. The photograph proves that the crystal has now transformed to the high form and therefore the correct energy reading for the transformation temperature lies between readings 3 and 4. This process can be continued so that this energy difference is refined as close as desired within the range of constancy of the furnace temperature and within the temperature hysteresis of the transformation.

When several such points have been determined, a smooth curve may be drawn relating the electrical input of the furnace to its temperature rise, called  $\Delta T$  in Fig. 9.  $\Delta T$  is the temperature rise above the cooling water temperature (which form a base level for temperature rises). The base level temperature is measured by means of a thermometer in the cooling water inlet, Fig. 10.

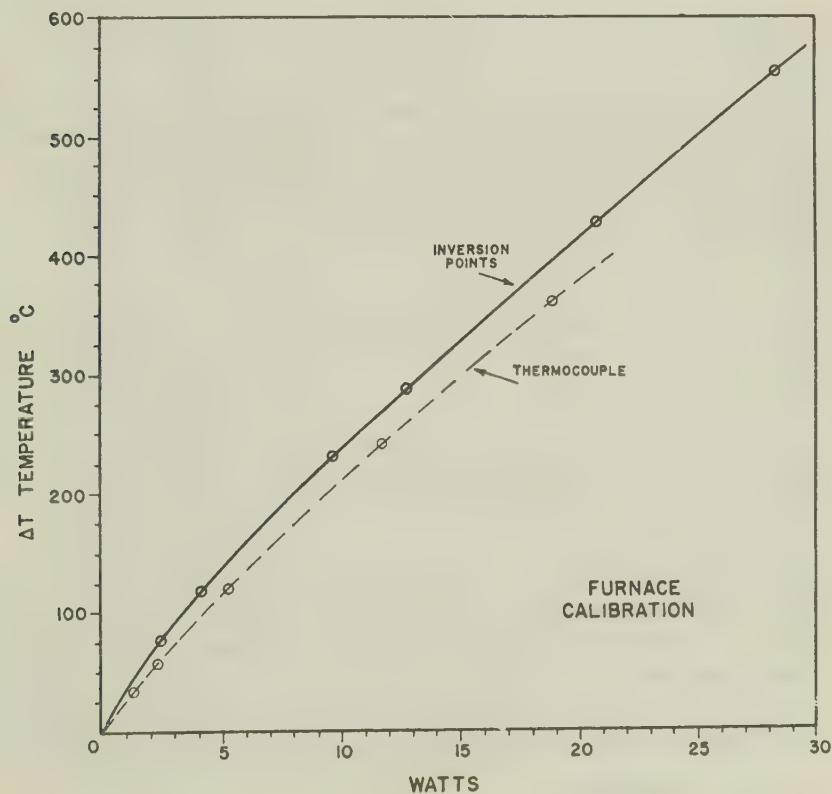


FIG. 9

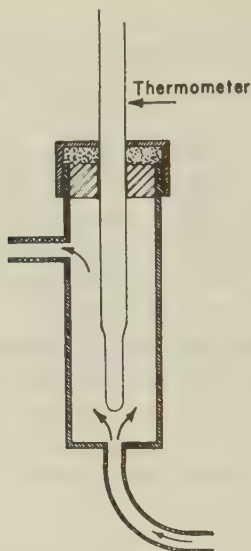


FIG. 10

Mr. Joseph Lukesh<sup>25</sup> has found empirically that if this relation is plotted on log-log graph paper, it can be represented by a straight line. This greatly aids in both interpolation between calibrated values and extrapolation. The relation between input voltage to the furnace and temperature rise has the general form:

$$\Delta T = kV^a.$$

The approximate values for the powder camera here described and the Weissenberg camera mentioned beyond are:

Powder camera

$$\Delta T \cong 22 V^{1.17}$$

Weissenberg camera

$$\Delta T \cong 10.5 V^{1.71}$$

A rough analysis of the relation between temperature and power input for the two furnaces shows that:

Powder camera

$$\Delta T \propto P^{0.60}$$

Weissenberg camera

$$\Delta T \propto P^{0.85}$$

If the heat losses were completely due to radiation, then  $\Delta T \propto P^{0.25}$ , assuming the furnace temperature is considerably higher than that of the surroundings; and if they were completely due to conduction then  $\Delta T \propto P^{1.0}$ . The furnace in the Weissenberg camera evidently has a greater conduction loss than that in the powder camera. This can be accounted

<sup>25</sup> Personal communication.

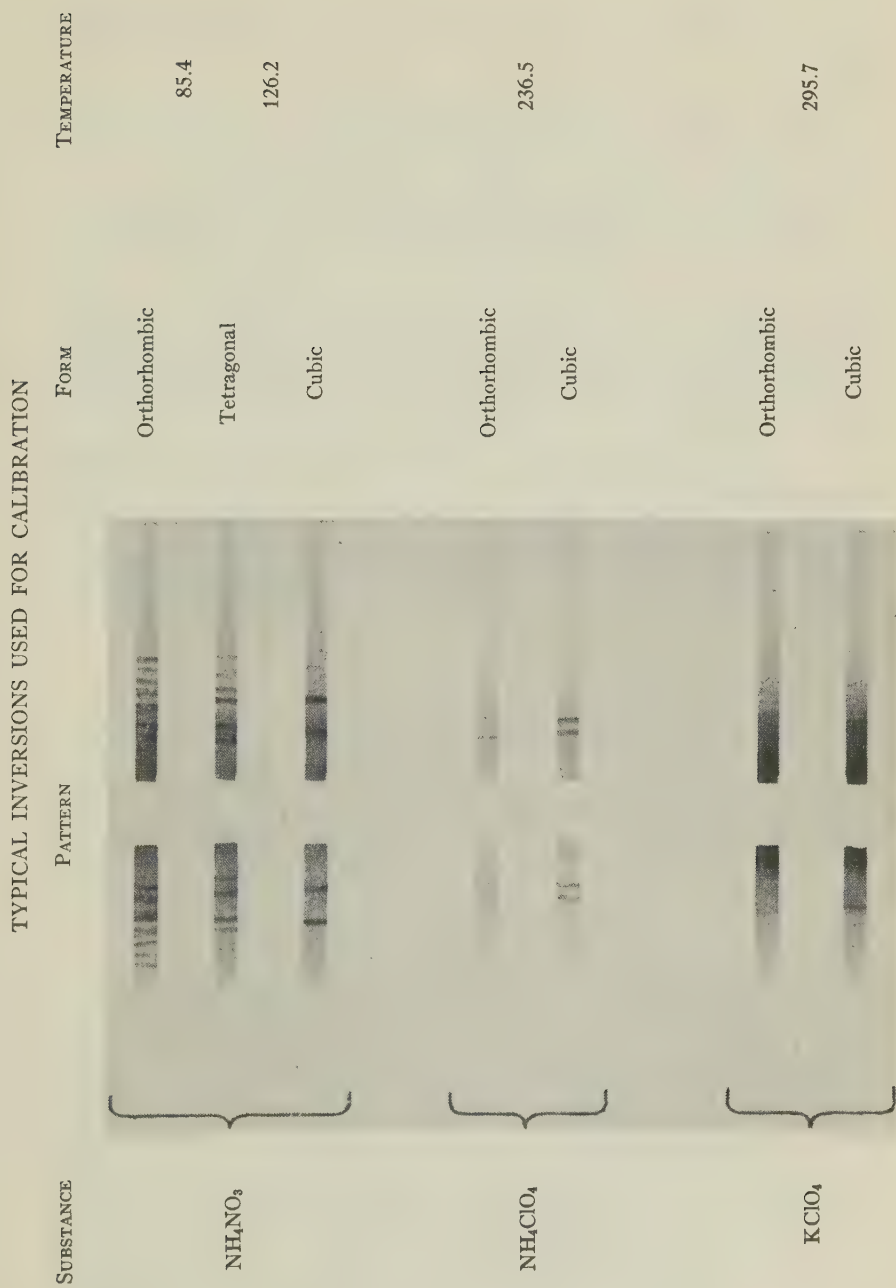


FIG. 11

for by the presence of a wider slot in the Weissenberg camera to accommodate facilities for equi-inclination procedures, other things being equal.

X-ray patterns of certain desirable substances showing polymorphism of a character useful in calibration are shown in Fig. 11. To this list may be added quartz, whose high-low transformation occurs at 573° C.

*Adaptation to Weissenberg Technique.* The above described heating unit with slight modification has been adapted to a Weissenberg<sup>26</sup> camera. In this adaptation, the heating chamber is built into a tube which slips over the spindle housing of the Weissenberg. This arrangement in no way interferes with the normal flexibility of the Weissenberg and it has been successfully used in the study of high chalcocite<sup>27</sup> and high tridymite.<sup>28</sup> It is hoped that a more detailed description of this apparatus will be published subsequently.

<sup>26</sup> Buerger, Newton W., Weissenberg controlled-temperature technique: *Am. Mineral.*, **27**, 217 (1942).

<sup>27</sup> Buerger, M. J., and Buerger, Newton W., Structural relations between high- and low-chalcocite: *Am. Mineral.*, **27**, 216 (1942).

<sup>28</sup> Buerger, M. J., and Lukesh, Joseph S., The tridymite problem: *Science*, **95**, 21 (1942).

# IRIDESCENT GARNET FROM THE ADELAIDE MINING DISTRICT, NEVADA

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## ABSTRACT

Garnets from a lime-rich layer in the contact zone of a granodiorite stock near Golconda, Nevada, show brilliant iridescence both on striated crystal faces and in thin section. They have a birefringence a little more than a third of that shown by orthoclase ( $0.0025 \pm$ ) and show, superposed on the triangular segments that are common in lime contact garnets, lamellae that look like polysynthetic twinning. Universal stage measurements show that the lamellae are parallel to (110) and (111). It appears that the iridescence is due to the very fine (111) lamellae and that it is more intense where the individual lamellae are finer. Sections of the garnets heated in a furnace show a decrease in birefringence beginning at about  $1060^{\circ}$  C. and continuing practically to the melting point just below  $1250^{\circ}$  C. In thin sections that have been heated almost to the melting point ( $1225^{\circ}$  C.) the birefringence is very low, but the twinning lamellae and iridescence are still visible.

## INTRODUCTION

Approximately one mile east of the abandoned Adelaide mining district, ten miles south of Golconda, Nevada, a granodiorite stock intruded shales, sandstones, and carbonate rocks of Permo-Triassic (?) age to form a varied series of hornfels and calc-silicate rocks (Fig. 1). At the south margin of the intrusion a carbonate bed was changed into a mass of dark honey colored to dark brown garnet.

## DESCRIPTION OF THE GARNET

Much of the garnet is in massive form, but many crystals are found at the surface outcrop and in an abandoned prospect pit. Crystals are rarely over two centimeters in diameter, more often under half a centimeter. Some are dodecahedrons; some are dodecahedrons modified by the trapezohedron; but most of the garnet occurs in crystals with irregularly shaped, mutually interfering faces which are striated in a manner similar to that seen on some pyrite crystals (Fig. 2). The garnet ranges in specific gravity from 3.50 for the light honey colored variety to 3.64 for the dark brown material. No complete quantitative analysis was made, but CaO and  $\text{Fe}_2\text{O}_3$  were determined in several specimens. The former runs as high as 23 per cent and the latter up to 28.5 per cent. Some

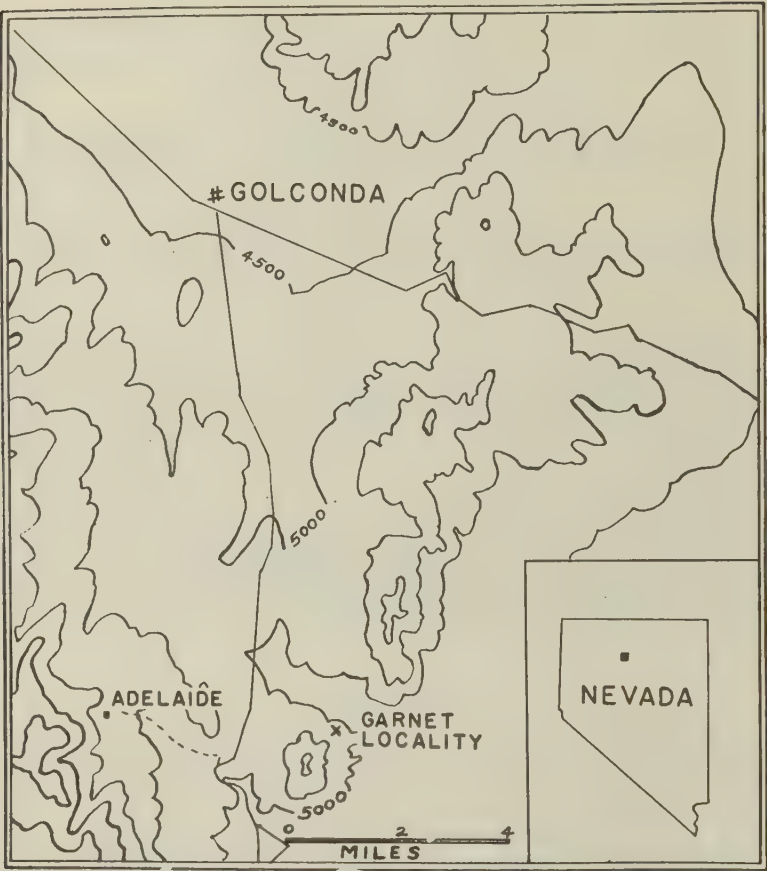


FIG. 1. Index map showing location of garnet locality.

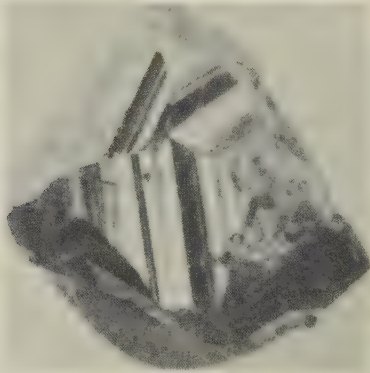
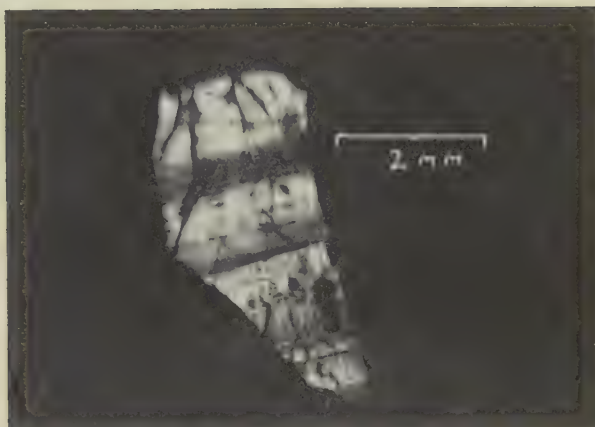


FIG. 2. Garnet crystal with striations.  $\times 3$ .



A Crystal face showing color banding parallel to striations.  $\times 10$ .



B. Thin section by reflected light.  $\times 10$ .

PLATE I. Iridescence in garnets from Nevada.



ferrous iron is present. The tests for magnesium were negative. These data, together with a rough index determination of 1.81, indicate that the garnet has more of the grossularite than the andradite molecule, and a little almandite.

The most striking feature about the garnet is the brilliant play of colors brought out by reflected light on the striated compound faces of most of the lighter colored crystals. The colors red, blue, green, orange, and yellow occur in bands parallel to the surface striations and are in places interrupted by interference colors due to thin air films in random cracks. The angle of incidence of the reflected light giving the most brilliant colors varies with each compound face. The color phenomenon is not due to a surface film; thin sections cut approximately parallel to the faces sharpen the color bands and eliminate the effects of random cracks. Plate 1 shows this iridescence; A, on a crystal face and B, in thin section.

Under the microscope the garnet is birefringent. Three types of structure are seen in thin section under crossed nicols: (1) A well defined banding which resembles twinning in plagioclase feldspars occurs parallel to the external striations (Fig. 3). This well defined banding is commonly confined to the peripheral zone of the crystals. The bands are dark when parallel to the vibration planes of the nicols. The peripheral zone is missing entirely in one of the thin sections; if it was present on the original crystal it was lost during grinding. One section, on the other hand, shows hardly any core (Fig. 3). Figure 4 shows this same crystal in parallel light.

Universal stage measurements indicate that the outer banding is parallel to the dodecahedron (110). The banding is relatively coarse, individuals ranging from 0.02 mm. to 1.7 mm. in thickness in the sections studied. Only rarely do these outer zones show any play of colors. When they do show it the color bands are always parallel to the outer banding, and upon closer study the latter are seen to have superposed upon them another much finer structure, (2), "herringbone" lamellae that universal stage measurements show to be parallel to the octahedral planes (111). This structure is shown in Fig. 5, which was taken at a higher magnification from the upper right hand corner of the crystal shown in Fig. 3. This appears to be true polysynthetic twinning; adjacent lamellae extinguish differently and alternate lamellae together. These lamellae do not have a uniform orientation throughout an entire crystal, or even in a given block of a segmented crystal, since they are developed parallel to more than one set of octahedral planes.

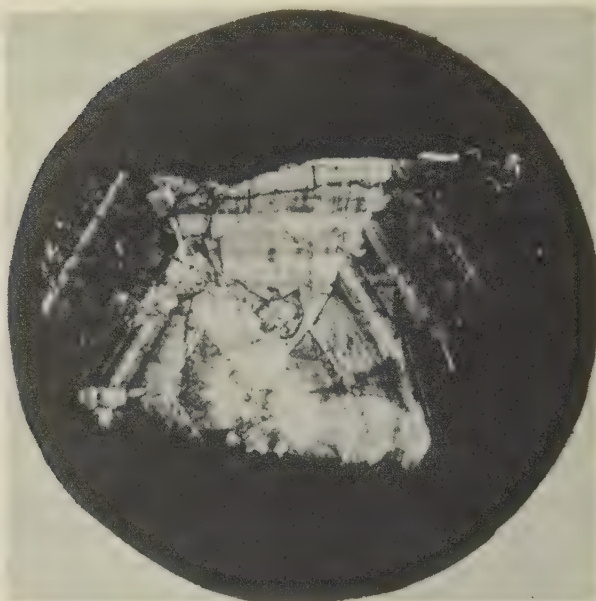


FIG. 3. Crystal in which the peripheral banding parallel to (110) is dominant. Crossed nicols.  $\times 10$ .



FIG. 4. Crystal of Fig. 3 in plane light.

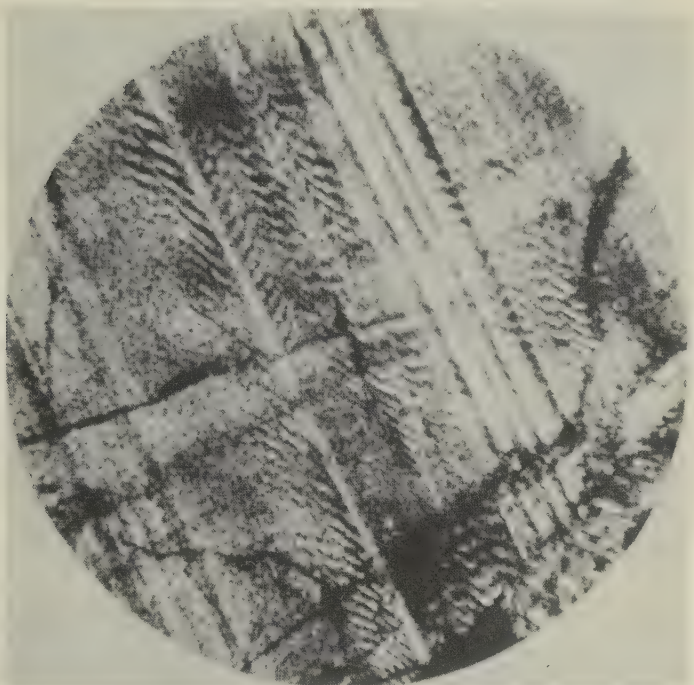


FIG. 5. Fine octahedral lamellae superposed on the dodecahedral lamellae. Crossed nicols.  $\times 100$ . From the upper right hand segment of the crystal shown in Fig. 3.

The highly iridescent cores of the garnet crystals show this fine twinning well developed and there appears to be a general correlation between twinning and iridescence. The cores show another structure; (3) a complex mottling not unlike the twinning in some varieties of microcline (Fig. 8). It is most pronounced in the  $45^\circ$  position, while in the parallel position a faint banding in the core parallel to the peripheral banding is seen. The color bands are parallel to this direction. There is no complete extinction of the mottled areas. That the mottling may be another aspect of the octahedral lamellae is indicated by an intimate association of the mottled and herringbone patterns. The mottling is too vague and the lamellae so broad that they cannot, in general, be sharpened up and measured accurately on the universal stage. This is probably due to the angle at which they are cut by the thin section, since some of them can be measured accurately and prove to be parallel to octahedral planes.

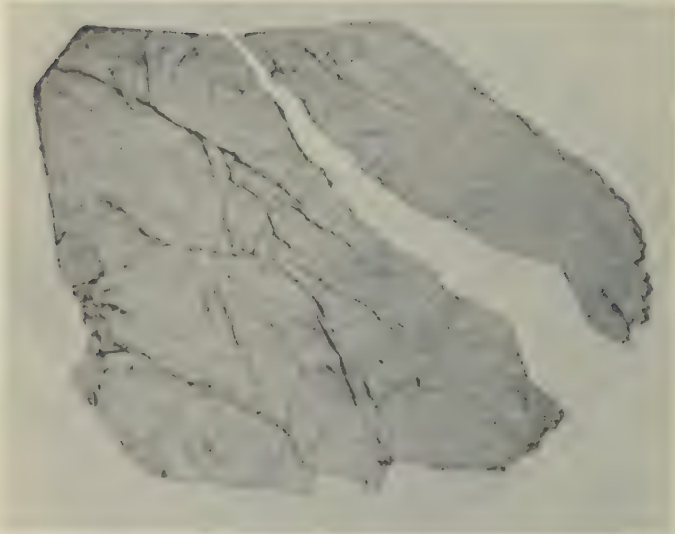


FIG. 6. Crystal of Plate I, B, as seen in plane transmitted light.  $\times 8$ .

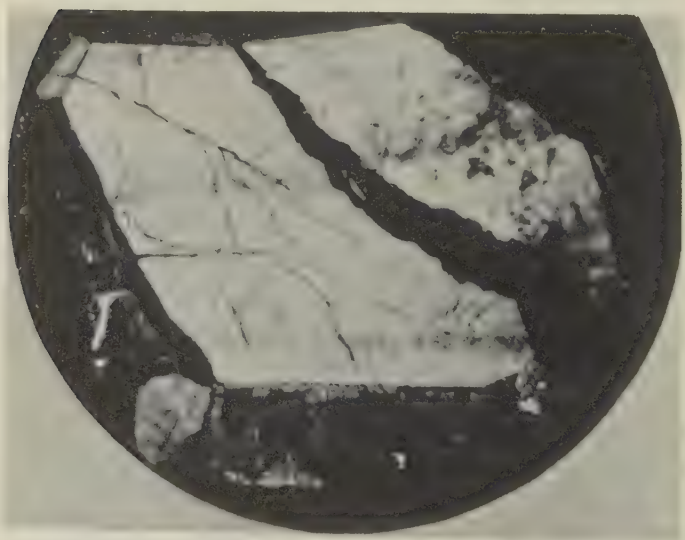


FIG. 7. Crystal of Fig. 6 between crossed nicols, showing strong peripheral (110) banding and faint banding in the core.  $\times 8$ .

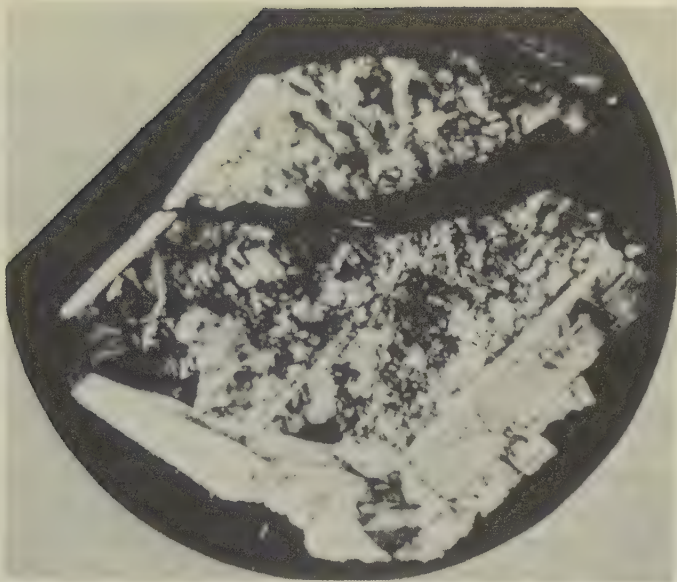


FIG. 8. Crystal of Fig. 6 in the  $45^\circ$  position between crossed nicols, showing the mottling of the core.  $\times 8$ .

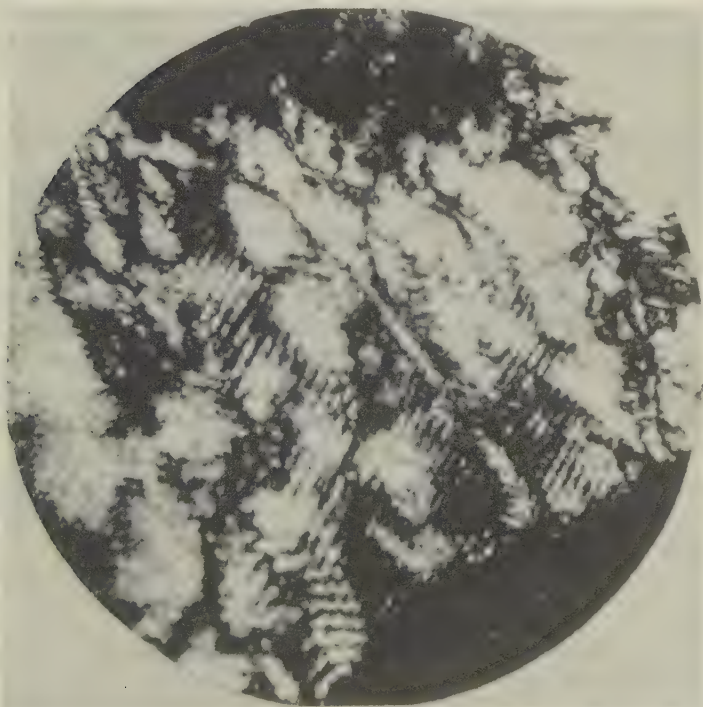


FIG. 9. Relation of mottling and octahedral lamellae. From lower edge of the smaller fragment of the crystal shown in Figs. 6-8. Crossed nicols.  $\times 100$ .

The relations of these various structures are shown in the photomicrographs. Figure 6 shows the crystal of Plate 1, B, as seen by plane transmitted light, the angle of incidence being such that the iridescence does not show. Figure 7 is the same crystal between crossed nicols and shows the sharp peripheral banding and the faint bands in the core parallel to the outer banding. Figure 8 is the same crystal in the  $45^\circ$  position, where the complex mottling obscures the banding in the core. Figure 9 is taken at a much higher magnification from the lower part of the smaller fragment of the same crystal and shows the relation of the fine octahedral lamellae to the mottling, as seen in one plane. Figure 5 shows the relation of dodecahedral and octahedral lamellae in the crystal shown in Figs. 3 and 4.

The universal stage measurements were made on sections normal to a dodecahedral crystal face, on sections approximately parallel to such faces, and on sections of unknown orientation. From each section several sets of lamellae were measured and the poles to the planes plotted on a stereographic projection and the angles between the various poles measured. The only advantage of oriented sections is that the orientation of one crystallographic plane is known and can be used for reference, whereas with the unoriented sections the identity of all lamellae has to be inferred from the angles measured. The answer was the same, of course, and probably was as trustworthy with the unoriented as with the oriented sections.

Twinning and birefringence are characteristic of contact garnets of grossularitic composition.<sup>1,2,3,4</sup>

The temperature at which such garnets become isotropic has been taken as an indication of the temperature below which they must have formed. Merwin<sup>5</sup> found, for example, that some contact garnets from Alaska lost their birefringence when they were heated to about  $800^\circ$  C. for a few hours, and did not regain it after several hours heating at  $600^\circ$ .

Similar experiments were performed with iridescent fragments and thin sections of the Nevada garnets. The thin sections were removed from the glass slides by heating and the balsam was removed by careful washing with xylol. They were heated on a piece of platinum foil and then transferred to a clean slide for examination. Each heating was continued for at least twelve hours. When any change was observed it took place in considerably less time than this.

<sup>1</sup> Schaller, W. T., Personal communication.

<sup>2</sup> Rogers and Kerr, *Optical Mineralogy*, 2nd ed., Fig. 284, p. 302.

<sup>3</sup> Wright, C. W., *U.S.G.S. Prof. Paper 87*, p. 50.

<sup>4</sup> Brauns, R., *Die optischen Anomalien der Kristalle*, Leipzig (1891) pp. 243-252.

<sup>5</sup> *U.S.G.S. Prof. Paper 87*, p. 108.

No change in the garnet was observed below 1060° C. At that temperature the birefringence weakened somewhat, but maintained the same pattern. Iridescence was as strong as ever. Reddish tinges began to appear, as if some  $\text{Fe}_2\text{O}_3$  was separating. At 1100° no further change occurred, but at 1185° the birefringence was still weaker. The octahedral lamellae were still visible in the center and the dodecahedral lamellae in the outer zones. Iridescence was still strong. At 1225° much of the garnet had broken up into spinel and pseudo-wollastonite, especially around the little stringers of calcite, but the general pattern and iridescence were unchanged. In the part that remained clear the octahedral and dodecahedral lamellae were clearly visible, but the birefringence was very weak. At 1250° the sections melted to a dark brown glass.

#### CAUSE OF THE PLAY OF COLORS

The play of colors in labradorite has been ascribed by Dana<sup>6</sup> (after Vogelsang) to a combination of lamellar structure and fine inclusions. The common peacock blue color is regarded as due to lamellar structure; the golden or reddish schiller as due to minute inclusions, or "to the combined effect of these with the blue reflections."

Kraus and Slawson<sup>7</sup> believe that the colors in labradorite may be due to reflections from fine inclusions or to fine twinning. This is essentially the same explanation given above from Dana.

Brögger<sup>8</sup> ascribed the play of colors in soda orthoclase to a cryptoperthitic development of the feldspar. Later work with  $x$ -rays<sup>9</sup> has borne out Brögger's deduction.

The play of colors in opal has been investigated by Baier<sup>10</sup> who concludes that the phenomena are due to interference of a series of reflections from internal faces arranged in hexagonal symmetry, inherited from a calcite residue in the primordial silica gel. He mentions also the development of ultra-microscopic twin lamellar structure.

Lord Rayleigh<sup>11</sup> has shown that the iridescence of many crystals of

<sup>6</sup> Dana, J. D., *System of Mineralogy*, 6th edition, John Wiley & Sons, Inc., New York (1892), p. 334.

<sup>7</sup> Kraus, E. H., and Slawson, C. B., *Gems and Gem Materials*, Third edition, McGraw-Hill Book Company, New York (1939), p. 216.

<sup>8</sup> Brögger, W. C., *Die Mineralien der Syenitpegmatitgänge der südnorwegischen Augit und Nephelinsyenit*: *Zeits. Kryst.*, **16**, 540–551 (1890).

<sup>9</sup> Kozu, Sh., and Endo, Y., *X-ray analysis of adularia and moonstone, and the influence of temperature on the atomic arrangement of these minerals*: *Sc. Rep. Tōhoku Imp. Univ.*, Series 3, **1**, No. 1, 1–17 (1921). This paper is abstracted in *Zeits. Krist.*, **57**, 116 (1922).

<sup>10</sup> Baier, E., *Optik der Edelopal*: *Zeits. Krist.*, **81**, 183–218 (1932).

<sup>11</sup> Lord Rayleigh, *Phil. Mag.* (5) **26**, 256 (1888); also, *Iridescent crystals*, Royal Institute Lecture, April 12, 1889. This theory is outlined in F. Pockels, *Lehrbuch der Kristalloptik*, Leipzig (1906), pp. 207–208.

potassium chlorate can be explained as due to reflections from a system of many very fine twinning lamellae of approximately equal thickness.

In the garnets described above there are no inclusions in the composition plane of the twinning that show up even under the highest power available (about 1350 $\times$ ). There are no visible cleavage planes parallel to the fine octahedral lamellae, and the fractures are too coarse and too irregular to explain the colors.

It appears, therefore, that the play of colors is due to reflection and interference phenomena related to the fine twin lamellae. This is indicated by the association of color bands and octahedral lamellae. The relation between iridescence and size of lamellae tends to confirm this opinion. The finer the lamellae the more intense the play of colors. In the brightest band seen in any section, the "herringbones" average 0.004 mm. in thickness. Where they are 0.006 mm. the colors are well developed, weaker at 0.0075, little or no color at 0.012, and colors were not found when the thickness of lamellae as seen in thin section exceeded 0.015 mm.

In one section each end shows color bands, but the central band shows no color. Under the microscope the central band shows mottling that is about twice as coarse as that of the two ends. The ends show the typical herringbone structure, which was not observed in the central band. In some cases this apparent difference in thickness of the lamellae, and the difference in iridescence, are due to different orientations of different parts of the crystal. This is true of Section W (Fig. 3), which is composed of six segments. However, the banded section referred to above is in uniform orientation, and the differences in lamellae thickness and iridescence are real.

It is concluded, therefore, that the play of colors in these garnets is due to very fine polysynthetic twinning, and that the intensity of the iridescence is dependent upon the thickness of the individual lamellae.

# RULES FOR THE CONVENTIONAL ORIENTATION OF CRYSTALS

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## ABSTRACT

The following rules are recommended for the conventional orientation of a crystal. They are intended to apply to all systematic descriptions, either morphological or structural.

1. The cell chosen to express *the* lattice should be the smallest cell having full lattice symmetry; to be defined by the shortest three non-coplanar translations, unless otherwise prescribed by the symmetry.

2. The axial cross should be right-handed.

3. The cell edges should be named  $c$ ,  $a$ ,  $b$  so that  $c < a < b$ , unless otherwise prescribed by the symmetry (in the monoclinic system, *e.g.*, the rule becomes  $c < a$ ).

4. The coordinate axes should be directed by the following conventions: (a) In the triclinic system,  $\alpha$  and  $\beta$  obtuse; (b) in the monoclinic system,  $\beta$  obtuse. Letting  $\phi(010) = 0$ , these conditions are respectively equivalent to: (a)  $\phi(001) < \phi(100)$ ; (b)  $\phi(001) = \phi(100) = 90^\circ$ .

Alternative rules ( $a < b < c$ , with  $\beta$  and  $\gamma$  obtuse; or  $b < c < a$ , with  $\gamma$  and  $\alpha$  obtuse) are provided for special problems, where the standard rules ( $c < a < b$ , with  $\alpha$  and  $\beta$  obtuse) would prove unsuitable.

Rules for the triclinic system, recently proposed by others, are discussed. The rule " $\alpha, \beta, \gamma$  all obtuse" contains a superabundant condition and hence cannot be applied in all cases. The rule " $\phi(001) < 90^\circ$ " is less natural than " $\phi(001) < \phi(100)$ ."

## INTRODUCTION

In 1933 Mélon and I published an attempt to systematize the conventions for the orientation of a triclinic crystal. The proposed rules were restated in a more precise form, shortly afterwards, in a joint paper (Donnay, Tunell, and Barth, 1934), which also included rules for the orientation of monoclinic and orthorhombic crystals. At that time the distinction had to be drawn between the *morphological lattice*, for which the Law of Haüy-Bravais held true, and the *structural lattice*, which defined the true periodicity of the crystal architecture as revealed by  $x$ -ray diffraction. The two lattices were not in agreement for all crystalline species, and it was recognized that different sets of conventional rules might have to be used in morphology and in structure. The rules proposed at that time were intended for morphological descriptions only.

Since then progress has been made in the field of the relationships between external form and internal structure of crystals. Present day morphological methods of crystal analysis usually lead to a lattice strictly proportional to the structural lattice; in favorable cases the latter can even be predicted in absolute dimensions, provided the chemical formula

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and the density of the compound be known. In other words, the two concepts of *morphological lattice* and *structural lattice* have now been unified—there is only one lattice, the same one for both the morphologist and the leptologist. These concepts, which had been readily accepted in mineralogical circles, may have already outlived their usefulness. A single set of rules for the conventional orientation of crystals, to be used in structural as well as in morphological descriptions, now appears extremely desirable.

### THE CRYSTAL LATTICE

Morphologists seem to have rallied to the idea that the crystal lattice should provide the coordinate system to be used—the lattice, that is to say the expression of the crystalline tri-periodicity. Except for very special reasons, a former *morphological lattice* will usually be discarded if different from that obtained by  $x$ -rays. Conversely, structural results in disagreement with morphological findings should be scrutinized anew, and either confirmed or invalidated.<sup>1</sup>

### THE CHOICE OF THE UNIT CELL

The first condition imposed on the unit cell chosen to define the lattice is that it should possess the full symmetry of the lattice. This condition must be considered in all systems except the triclinic.

The second rule is to choose the smallest cell that fulfills the first condition. Exceptions to this rule are as follows: In the monoclinic and orthorhombic systems, the Miller cell is used rather than the Lévy cell;<sup>2</sup> in the hexagonal system, a rhombohedral lattice should preferably be referred to the  $R$ -centered hexagonal cell.<sup>3</sup>

Morphologists, I believe, would be prepared to adhere to this rule, even though it may entail the reorientation of numerous minerals as, for exam-

<sup>1</sup> This has happened. Tourmaline was first assigned a simple hexagonal lattice on the basis of  $x$ -ray work, in contradiction to remarkable morphological extinctions that proclaimed the  $R$ -centering; on re-examination, the error was discovered (Buerger and Parish, 1937). A mistake in the determination of the unit cell of aramayoite by  $x$ -ray work was found by morphological analysis (Berman and Wolfe, 1939). In the case of stephanite an aberrant spot on a Weissenberg picture had led to a space-group different from that indicated by the good morphological development of the crystals; additional photographs confirmed the morphological result (Taylor, 1940). The polemics about chalcopyrite too could have been settled by the morphological evidence, which leads to the correct indices  $\{112\}$  for the pseudo-tetrahedron, long erroneously symbolized as  $\{111\}$ .

<sup>2</sup> In other words: The  $b$  axis of a monoclinic crystal must be the 2-axis of the lattice; the  $c$  and  $a$  axes must lie in the plane of symmetry of the lattice. The cell edges of an orthorhombic crystal must be the 2-axes of the lattice.

<sup>3</sup> A statement of the rhombohedral axial elements should, however, accompany the description in terms of the  $R$ -centered hexagonal cell.

ple, in the tetragonal system, where body-centered species were often described in the face-centered setting.

As to structural crystallographers, one will notice that the *International Tables* (1935) make an exception to this rule in the case of crystals belonging to the class  $\bar{4}2m$ , the multiple cell being preferred whenever necessary to avoid the orientation  $\bar{4}m2$ . Thus, their conventional setting<sup>4</sup> of the space group  $D_{2d}^5$  is not  $P\bar{4}m2$ , but  $C\bar{4}2m$ ; likewise,  $D_{2d}^9$  is not written  $I\bar{4}m2$ , but  $F\bar{4}2m$ . The same policy is again followed in classes  $\bar{6}m2$ ,  $\bar{3}2/m$ ,  $32$ ,  $3m$ , in order to obviate the necessity of using the orientation  $\bar{6}2m$ ,  $\bar{3}12/m$ ,  $312$ ,  $31m$ . This probably was a concession to some old conventions governing the setting in such classes. They may well be discarded now, for the sake of uniformity, as was done by Buerger in his recent book (1942).

In cases where the cell is not imposed by the symmetry (triclinic and monoclinic systems), the cell with the shortest translations is chosen. This convention offers no disadvantage whatsoever in the triclinic system, where it seems to be universally accepted. In the monoclinic system, the rule must apply only to the choice of the  $c$  and  $a$  axes. It may conflict with two conventions of the *International Tables*, which in case of centering propose to make the lattice base-centered ( $C$ ), and in case of glide advise to make the glide-direction the  $c$  axis. The advantages of the rule of the shortest translations should outweigh all other considerations in most instances. Examples of difficulties have been encountered in sylvanite, realgar, lorandite, lanarkite (see appendix).

#### NAMING THE AXES

The naming of the axes is imposed by the symmetry of the lattice, in all except the trimetric systems. Two axes ( $c$ ,  $a$ ) in the monoclinic system and all three axes ( $c$ ,  $a$ ,  $b$ ) in the triclinic and orthorhombic systems need be conventionally labelled.

The time-honored morphological rule was to set the axis of the *main zone* vertical (making it the  $c$  axis). It must be abandoned, at least in that form, as it cannot be applied by structural workers who may be dealing with crystals devoid of faces. The concept of the main zone always lacked clarity; it has been construed to mean a zone of elongation, a zone perpendicular to a tabular habit, a zone rich in faces, etc. Donnay, Tunell, and Barth (1934) defined it as the direction of elongation in all cases except that of tabular orthorhombic crystals. Peacock (1937a) added another exception, namely that in tabular, pseudo-dimetric crystals, the plane of flattening should become the base (001).

<sup>4</sup> For the conventional orientation of the *International Tables*, the space-group symbol is shown in italics in Donnay and Harker's *Tables* (1940).

Clearly, if the new conventional rules are to be adopted by structural crystallographers, they must be expressed in terms of lengths of axial translations. Naumann's old rule to take  $a < b$ , first proposed for orthorhombic crystals, is now widely accepted for triclinic crystals as well; it should be retained, but it does not suffice. According to the Law of Bravais,<sup>5</sup> if a crystal is set with its direction of elongation vertical,  $c$  should be the smallest translation. The morphologists' rules as to the naming of the axes can thus be interpreted as:  $c < a < b$ . This convention is applicable to both the triclinic and orthorhombic systems, if the provisos concerning tabular habits be abandoned. In the monoclinic system, the rule reduces to  $c < a$ .

The monoclinic rule ( $c < a$ ) was proposed by Barker (1930), albeit for other purposes, and by Donnay, Tunell, and Barth (1934, p. 446). The rule  $c < a < b$ , proposed by Peacock (1937*a*, p. 596) for usually elongated triclinic crystals, has been used in orthorhombic cases too. In fact this convention has been rather generally adopted by mineralogists in the past five years; Buerger (1942, p. 366) admits that the custom "prevails."

Buerger (1942) proposes to replace the prevailing custom ( $c < a < b$ ) by the rule  $a < b < c$ . His case against the convention  $c < a < b$  is not convincing: (1) He writes (p. 367) that the ultimate explanation of crystal habit depends upon packing and bonding of atoms rather than identity periods. I do not wish to question this statement, but it so happens that, in most cases, the packing and bonding explanation coincides with that based on identity periods.<sup>6</sup> (2) He says (p. 366) that "though it is true that the choice of labels is arbitrary, the same arguments can be advanced against the choice of this order ( $c < a < b$ ) as against the choice of the order  $\gamma < \alpha < \beta$  for refractive indices." The question then becomes one of pure formalism. While it is true that the ordinal arrangement is very simple, it may be pointed out that any one of the three cyclic permutations ( $cab$ ,  $abc$ ,  $bca$ ) is just as elegant as any other; in fact the rule  $c < a < b$  could be preferred, on formalistic grounds alone, because it reduces to  $c < a$  in the monoclinic system.

<sup>5</sup> This aspect of the Law of Bravais is not commonly known, or appreciated: a crystal is usually elongated parallel to the lattice row of highest *linear density* (number of nodes per unit of length on the row). A statement of this law may be found in Mauguin's book on crystal structure (1924).

<sup>6</sup> Although the Law of Bravais is not perfect and, even in its generalized form, remains susceptible of refinements, it nevertheless embodies a mass of observed facts which—as such—cannot be lightly dismissed. Friedel's imposing evidence (1904, 1907) established the empirical character of the law, experimentally valid regardless of any of the speculations that led Bravais to formulate it. The accumulated x-ray data on crystals have confirmed the relation of habit to lattice periods in the majority of cases.

## DIRECTING THE AXES

Most modern crystallographers use the right-handed system of coordinates; the axial cross being conventionally so oriented that the  $c$  axis is positive upward, the  $a$  axis toward the observer, the  $b$  axis to the right of the observer.

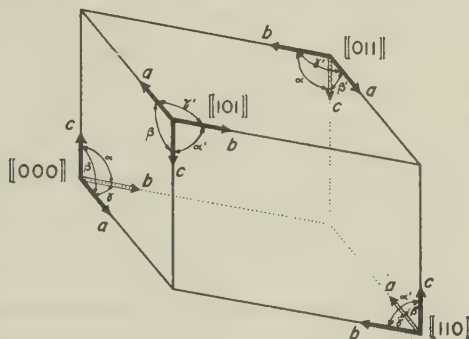


FIG. 1. The four ways of directing the named axes so as to obtain a right-handed axial cross.

A triclinic cell having been chosen, and its axes named (according to any convention) but not directed, there are four ways of choosing a right-handed system of coordinates. This can be visualized (Fig. 1) by placing axial crosses at the cell corners  $[000]$ ,  $[011]$ ,  $[101]$ ,  $[110]$ . These four axial crosses differ by the values of their interaxial angles  $\alpha$ ,  $\beta$ ,  $\gamma$  (respectively between the axes  $bc$ ,  $ca$ ,  $ab$ ). If at any corner of the cell, say  $[000]$ , the interaxial angles are  $\alpha$ ,  $\beta$ ,  $\gamma$ ; then they will be  $\alpha$ ,  $\beta'$ ,  $\gamma'$ , at  $[011]$ ;  $\alpha'$ ,  $\beta$ ,  $\gamma'$ , at  $[101]$ ;  $\alpha'$ ,  $\beta'$ ,  $\gamma$ , at  $[110]$ ; where the primed letters designate supplementary angles ( $\alpha' = 180^\circ - \alpha$ ,  $\dots$ , etc.). At any corner, say  $[000]$ , the interaxial angles may have any values, in general different from  $90^\circ$ , acute or obtuse. All the possibilities are summarized in Table 1, where an acute angle is represented by a + sign and an obtuse one by a - sign.

The tabulation shows that only in one half of the cases (possibilities Nos. 1, 2, 3, 4) will one be able to find an axial cross with all three interaxial angles obtuse. In every case, on the other hand, the choice of the axial cross is uniquely determined by the condition that two stated angles, say  $\alpha$  and  $\beta$ , be obtuse. The only convention necessary for directing the axes (assuming a right-handed system of coordinates) is thus that the interaxial angles  $\alpha$  and  $\beta$  be both obtuse. This rule was recommended by Donnay, Tunell, and Barth in 1934. It can be variously stated: by saying that the  $a$  axis should slope to the front ( $\beta > 90^\circ$ ) and the  $b$  axis to the right ( $\alpha > 90^\circ$ ), or, more simply, that the base (001) should slope

TABLE 1. ACUTE OR OBTUSE CHARACTER OF THE INTERAXIAL ANGLES IN THE FOUR RIGHT-HANDED AXIAL CROSSES FOR EACH OF THE EIGHT POSSIBILITIES

| Possibilities | Corner [[000]] |         |          | Corner [[011]] |          |           | Corner [[101]] |         |           | Corner [[110]] |          |          |
|---------------|----------------|---------|----------|----------------|----------|-----------|----------------|---------|-----------|----------------|----------|----------|
|               | $\alpha$       | $\beta$ | $\gamma$ | $\alpha$       | $\beta'$ | $\gamma'$ | $\alpha'$      | $\beta$ | $\gamma'$ | $\alpha'$      | $\beta'$ | $\gamma$ |
| 1             | —              | —       | —        | —              | +        | +         | +              | —       | +         | +              | +        | —        |
| 2             | —              | +       | +        | —              | —        | —         | +              | +       | —         | +              | —        | +        |
| 3             | +              | —       | +        | +              | +        | —         | —              | —       | —         | —              | +        | +        |
| 4             | +              | +       | —        | +              | —        | +         | —              | +       | +         | —              | —        | —        |
| 5             | —              | —       | +        | —              | +        | —         | +              | —       | —         | +              | +        | +        |
| 6             | —              | +       | —        | —              | —        | +         | +              | +       | +         | +              | —        | —        |
| 7             | +              | —       | —        | +              | +        | +         | —              | —       | +         | —              | +        | —        |
| 8             | +              | +       | +        | +              | —        | —         | —              | +       | +         | —              | —        | +        |

N.B.—The character of an angle is indicated by the sign of its cosine: acute angle, +; obtuse angle, —.

forward and to the right of (100). In terms of the usual azimuthal angles of face poles, letting  $\phi(010)=0$ , it is also equivalent to the condition  $\phi(001)<\phi(100)$ .

Buerger proposes (1942, p. 366) to “take  $+a$ ,  $+b$ , and  $+c$  in such directions that the interaxial angles  $\alpha$ ,  $\beta$ , and  $\gamma$  are all obtuse.” The interesting corollary of this rule, he points out, is that “the interaxial angles  $\alpha^*$ ,  $\beta^*$ , and  $\gamma^*$ , of the reciprocal cell are acute.” The corollary is true, but the rule unfortunately implies a superabundant condition and, as has been shown above, will lead to no solution in 50 *per cent* of the cases. It must therefore be amended.

Other crystallographers in the past had proposed the same fallacious rule of three obtuse interaxial angles. Bauer (1886, p. 99) writes: “Man stellt die Krystalle gerne so auf dass die Winkel  $\alpha$ ,  $\beta$ ,  $\gamma$  im vorderen, oberen, rechten Oktanten stumpf sind.” Walker (1914, p. 137) says that “the crystal is usually so oriented that the obtuse angles formed by the three axes are all enclosed by the positive ends of the axes.” Niggli (1920, p. 50) records the usual practice that one axis ( $c$ ) is placed vertically, another ( $b$ ) sloping slightly from left to right, the third one ( $a$ ) sloping relatively slightly forward; and he adds: “Alle drei Achsen bilden dann hier im vorderen rechten  $+$ -Oktanten . . . stumpfe Winkel  $\alpha$ ,  $\beta$ ,  $\gamma$  miteinander.” Yet (p. 123) he gives axinite with  $\alpha$  acute and albite with  $\gamma$  acute.

In the matter of directing the axes, Peacock (1937*a*, p. 594) stated the rule that the base should slope front-right, in the belief (which I shared at the time) that it was equivalent to the condition  $\alpha$  and  $\beta$  obtuse. He

soon corrected the error (1937*b*), but decided to discard the rule in favor of the  $0 < \phi(001) < 90^\circ$  condition. Although this condition, equivalent to  $\beta$  obtuse and  $\alpha^*$  acute, does give a unique setting and has several advantages (simplicity of statement, easy recognition in gnomonic projection or on a model), it introduces into the triclinic system an artificial orthogonality concept and entails complicated consequences. Professor Peacock has now agreed<sup>7</sup> to revert to the rule  $\alpha$  and  $\beta$  both obtuse, equivalent to  $0 < \phi(001) < \phi(100)$ .

Hurlbut (1941, p. 57) adopts the rule (taken from Peacock) that the base should slope forward and to the right. If, after the words "to the right," be added "of the front pinakoid (100)," then the statement is brought in agreement with the convention  $\alpha$  and  $\beta$  both obtuse.<sup>8</sup>

It is of interest to recall that Barker (1930), in his work on goniometric determination of (non-isometric) crystalline substances, encountered the problem of determining a unique conventional orientation. For directing previously named axes in the triclinic system, he stated rules that are equivalent to taking  $\alpha$  and  $\beta$  both obtuse. Although, as Peacock pointed out (1937*b*), the Barker method was only intended as a determinative tool and not a general system of morphological descriptions, it remains true that when one reaches the stage of selecting arbitrary rules in order to attain a unique orientation (after a cell has been chosen and the axes named) no question of propriety from the morphological point of view is involved any more. It is then simply a matter of choosing rules on the sole merit of their convenience. The fact that Barker's choice was the convention " $\alpha$  and  $\beta$  both obtuse" is undeniably an argument in its favor.

Finally it remains to be stated that the rule for directing the axes reduces to  $\beta$  obtuse in the monoclinic system. This is equivalent to saying that the base should slope forward. In terms of the usual azimuthal angles of face poles, letting  $\phi(010) = 0$ , the rule is expressed:  $\phi(001) = \phi(100) = 90^\circ$ . This convention has long enjoyed universal recognition.

### CONCLUSIONS

From the foregoing considerations, it seems that the recommended set of conventional rules ( $c < a < b$ ;  $\alpha$  and  $\beta$  both obtuse) is probably as good as any other arbitrary set. It has already won wide acceptance among mineralogists.

<sup>7</sup> Private communication.

<sup>8</sup> Incidentally, note that axinite, given by Hurlbut to illustrate his rule, is still oriented as in the *Dana System* (1892), namely  $a:b:c = 0.492:1:0.480$ ,  $\alpha = 82^\circ 54'$ ,  $\beta = 91^\circ 52'$ ,  $\gamma = 131^\circ 32'$ . With these elements the base does not slope to the right, but slopes considerably to the left, as  $\phi(001)$  is approximately equal to  $166^\circ$ .

In certain special problems, however, it may happen that this set of rules becomes unsatisfactory; for example, in cases of unusual habit, pseudosymmetry, homeomorphism between related species, imperfect morphological development (in the absence of *x*-ray data), etc. It is well, therefore, not to make the rule too rigid. Any orientation that can be derived from the standard one (recommended above) by cyclic permutations of the unit-lengths and the interaxial angles, should prove acceptable in such special problems. The rules could be enlarged<sup>9</sup> as follows: The axes will be named so as to satisfy the condition  $c < a < b$ , or a cyclic permutation thereof (either  $a < b < c$ , or  $b < c < a$ ). In any case the axes will be directed so as to render obtuse the interaxial angles opposite the largest two unit-lengths. (If one angle has to be acute, it will always be that opposite the smallest unit-length.)<sup>10</sup>

The choice is thus limited to the three alternatives: (1)  $c < a < b$ ,  $\alpha$  and  $\beta$  obtuse; (2)  $a < b < c$ ,  $\beta$  and  $\gamma$  obtuse; (3)  $b < c < a$ ,  $\gamma$  and  $\alpha$  obtuse.

Posnjak and Tunell (1929) gave a morphological description of the compound  $3\text{CuO} \cdot 2\text{SO}_3 \cdot 5\text{H}_2\text{O}$ , in which the elements were

$$a:b:c = 0.7805:1:1.0760, \alpha = 103^\circ 4', \beta = 99^\circ 7', \gamma = 104^\circ 48',$$

thus obeying the rule  $a < b < c$ . (The lattice was not determined by *x*-rays; therefore the above elements do not necessarily express the true periodicity.) The zone placed vertically is a zone rich in faces; the crystals are somewhat equant, tabular on {010}. This is an example of a difficult case in finding the lattice by morphology alone.

Richmond (1942) has recently reoriented inesite, with the following elements:

$$a_0 = 8.89, b_0 = 9.14, c_0 = 12.14, \alpha = 87^\circ 38\frac{1}{2}', \beta = 132^\circ 30', \gamma = 97^\circ 5\frac{1}{2}',$$

which also illustrates the rule  $a < b < c$ . The transformation matrix 010/001/100 would lead to the elements:

$$a_0 = 9.14, b_0 = 12.14, c_0 = 8.89, \alpha = 132^\circ 30', \beta = 97^\circ 5\frac{1}{2}', \gamma = 87^\circ 38\frac{1}{2}',$$

directly comparable with the former (by cyclic permutation).

#### ACKNOWLEDGMENTS

My best thanks are due to the following gentlemen, with whom I discussed the conventional rules proposed in this paper: Dr. Harry Berman (Harvard University), Dr. M. J. Buerger (Massachusetts Institute of

<sup>9</sup> Professor Harry Berman has agreed (private communication) to follow these rules as far as possible in the work he is doing on the revision of the *Dana System*.

<sup>10</sup> Professor M. J. Buerger has agreed (private communication) that, if one angle must be acute, the angle opposite the smallest unit-length should be the acute one.

Technology), Dr. David Harker (General Electric Co.), Dr. W. J. Kirkpatrick (Hercules Experiment Station), Dr. M. A. Peacock (University of Toronto), Dr. Franco Rasetti (Université Laval), Dr. G. Tunell (Geophysical Laboratory).

The treatment of the four monoclinic species (Appendix I) is based on the work I did at Harvard University, during the summer of 1937, in connection with the revision of Dana's *System*. It is a pleasure to recall the fruitful discussions I had at the time with Professor Charles Palache and his co-workers.

## APPENDIX I

### MONOCLINIC CRYSTAL SYSTEM

#### *Sylvanite*

Schrauf (1878) chose the following axial elements:  $a:b:c = 1.6339:1:1.1265$ ,  $\beta = 90^\circ 25'$ , intended to bring out a remarkable orthorhombic pseudo-symmetry.

Friedel (1904*b*, p. 410) found that the lattice defined by the Schrauf elements would, provided it be centered on  $\{010\}$ , give the Law of Bravais an adequate expression.

The structural lattice (Tunell and Ksanda, 1937), however, can only be obtained by a halving of the  $b$  unit-length in addition to the  $\{010\}$ -centering. The  $x$ -ray investigation revealed the fact that the plane of monoclinic symmetry is a glide-plane, with glide-component in the direction of Schrauf's  $a$  axis. Following the recommendations of the *International Tables for the Determination of Crystal Structures*, Tunell and Ksanda adopted the direction of the glide-component as their  $c$  axis and chose the smallest translation in the  $(010)$  net as their  $a$  axis.

Peacock (unpublished) proposed to take the shortest translation in the  $(010)$  plane as the  $c$  axis and the next short one as the  $a$  axis ( $c < a$ ).

The relationships between the three settings are shown in a projection of the direct lattice on the  $(010)$  plane (Fig. 2) and in the transformation matrices (Table 2).

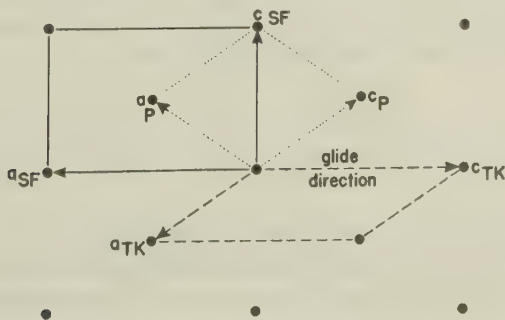


FIG. 2. Direct lattice of sylvanite projected on  $(010)$ . Solid line, Schrauf-Friedel; dashed line, Tunell-Ksanda; dotted line, Peacock. The  $b$  unit-length of Schrauf is twice that of the other two settings; the negative  $b$  of Tunell-Ksanda is the positive  $b$  of the other settings.

TABLE 2. TRANSFORMATION MATRICES FOR SYLVANITE SETTINGS

| From \ To                                               | Schrauf-Friedel         | Tunell & Ksanda                                             | Peacock                                                                             |
|---------------------------------------------------------|-------------------------|-------------------------------------------------------------|-------------------------------------------------------------------------------------|
| Schrauf-Friedel<br>( <i>B</i> -centered)<br>(1878-1904) | 1 0 0<br>0 1 0<br>0 0 1 | $\frac{1}{2}$ 0 $\frac{1}{2}$<br>0 $\frac{1}{2}$ 0<br>1 0 0 | $\frac{1}{2}$ 0 $\frac{1}{2}$<br>0 $\frac{1}{2}$ 0<br>$\frac{1}{2}$ 0 $\frac{1}{2}$ |
| Tunell & Ksanda<br>(1937)                               | 0 0 1<br>0 2 0<br>2 0 1 | 1 0 0<br>0 1 0<br>0 0 1                                     | 1 0 1<br>0 1 0<br>1 0 0                                                             |
| Peacock<br>(unpublished)                                | 1 0 1<br>0 2 0<br>1 0 1 | 0 0 1<br>0 1 0<br>1 0 1                                     | 1 0 0<br>0 1 0<br>0 0 1                                                             |

N.B.—This table gives face-to-face transformations, which also serve to transform lattice vectors (hence the fractional values of some of the matrix elements).

The influence of the glide-plane of symmetry on the morphology is expressed in the space-group criterion:

$$\{h0l\} \text{ with } l \text{ even,}$$

in the case of the Tunell-Ksanda setting ( $P2/c$ ). Twelve forms have been observed in that zone; they are:

$$\text{I01. I00. } \bar{2}02. 002. \bar{3}04. \text{I04. I02. } \bar{5}06. 202. \bar{4}02. \bar{5}04. \text{I04.}$$

Eight out of twelve already had  $l$  even without the artifice of the "multiple indices." The forms whose indices have to be doubled are either important forms, with very small indices, which remain among the first of the theoretical sequence even after the symbols are doubled ( $\text{I01}=\bar{2}02$ ,  $001=002$ ), or uncommon forms, which are appropriately made to recede in the theoretical list of decreasing importance ( $\text{I01}=\bar{2}02$ ,  $201=\bar{4}02$ ). The space-group restriction is seen to be remarkably obeyed. The setting of Tunell and Ksanda permits writing both the space-group symbol ( $P2/c$ ) and criterion ( $\{h0l\}$ , with  $l$  even) in the simplest way.

In the Peacock setting, the space-group criterion becomes

$$\{p0r\} \text{ with } (p-r) \text{ even.}$$

This is seen immediately from the transformation "Tunell-Ksanda to Peacock," since  $\{h0l\}$  to T.-K. becomes  $\{p0r\} = \{h+l. 0. h\}$ , in which  $l$  must be even. The space-group symbol must be written  $P2/n$ . The glide-component is  $\frac{1}{2}(\mathbf{c}+\mathbf{a})$ . The forms in the  $[010]$  zone are written:

$$\text{I01. I01. } \bar{0}02. 200. \text{I03. } \bar{3}01. \bar{3}01. \text{I05. } \bar{4}02. 204. \text{I05. } \bar{5}01.$$

The space-group symbol and criterion are a little less simply expressed than in the Tunell-Ksanda setting, but this is hardly a compelling reason for rejecting the conventions followed by Peacock.

In the Schrauf setting,  $\{h0l\}$  of Tunell-Ksanda becomes  $\{e0g\} = \{l. 0. 2h+l\}$ . This shows immediately that  $(e+g)$  must be even ( $B$ -centering criterion) and that  $e$  must be even (space-group criterion). It follows that  $g$  must also be even; so that, in the symbols  $\{e0g\}$ , all three indices must be even. Morphologically, this is the equivalent of no condition at

all, and the forms  $\{e0g\}$  should constitute a *simple zone*. They can be written as follows (all indices divided by 2):

$$100.201.101.203.102.103.001.\bar{1}03.203.\bar{1}01.302.201.$$

All other forms must obey the *B*-centering criterion:  $\{efg\}$  with  $(e+g)$  even.

### Realgar

The case of realgar is similar.

Goldschmidt (1904) referred it to a set of axial elements,

$$a:b:c = 0.7203:1:0.4858; \beta = 113^\circ 44',$$

which were later found to correspond to a unit cell of the structural lattice (Buerger, 1935).

The glide-component  $\frac{1}{2}a$  is directed along the *a* axis of Goldschmidt, and the space-group could be symbolized  $P2_1/a$  if the Goldschmidt cell were retained. Buerger prefers to select another unit cell, defined by the shortest two identity periods in the (010) plane. The translation [101] of Goldschmidt is smaller than his  $a[100]$  unit length; Buerger chooses it accordingly for his own *a* axis. The transformations are as follows: Goldschmidt to Buerger = Buerger to Goldschmidt =  $\bar{1}0\bar{1}/0\bar{1}0/001$ .

In Buerger's setting the glide-component is  $\frac{1}{2}(c+a)$  and the space-group is accordingly written  $P2_1/n$ .

The space-group criterion for  $\{h0l\}$  is "*h* even" in the Goldschmidt, " $(h+l)$  even" in the Buerger, setting. The conventional setting of the *International Tables* would make the glide direction that of the *c* axis, the space-group would be written  $P2_1/c$ , and the space-group criterion would be, for  $\{h0l\}$ , "*l* even." An additional criterion, for  $\{0k0\}$ , is common to all settings: "*k* even."

The relationship between the three settings is shown in Fig. 3.

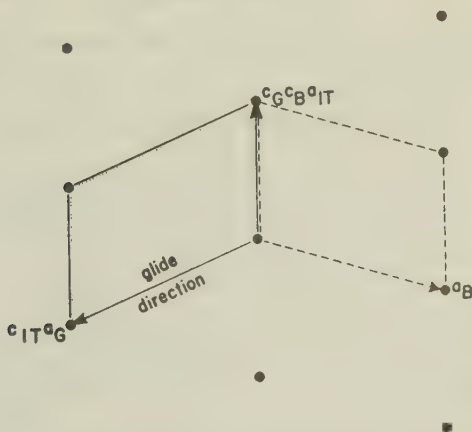


FIG. 3. Direct lattice of realgar projected on (010). Solid line, Goldschmidt; dashed line, Buerger; dotted line, *International Tables*. The negative *b* of Buerger is the positive *b* of the other settings.

Only five forms are known in the [010] zone. They are, in Goldschmidt's setting, 202.200.201.001.601; in Buerger's setting, 002.200.101. $\bar{1}01$ .501. Three out of the five obey the space-group criterion without multiplication of indices.

*Lorandite*

The case of lorandite is perhaps the most instructive of the four considered here.

Lorandite was referred by Krenner (1894) to a set of elements that bring out the orthorhombic pseudo-symmetry, namely:

$$a:b:c = 0.8534:1:0.6650; \beta = 90^\circ 17'.$$

Goldschmidt (1898) obtained a simplification of the indices by abandoning the pseudo-orthorhombic elements and choosing the following:

$$a:b:c = 1.3291:1:1.0780; \beta = 127^\circ 33'.$$

The half diagonal of Krenner's *ca* mesh became the new *c*, the negative *c* was made the new *a*, and the *b* unit length was halved. The new cell was not the smallest cell of the lattice, however, as later investigation showed.

Ungemach (1923, p. 155) proposed another set of axial elements, also chosen on morphological grounds, but which corresponded to the unit cell of the lattice, as was proved nine years later (Hofmann, 1932). Goldschmidt's *c* was halved and became the new *a*, the negative *b* was taken as the new *b*, and the new *c* was the vectorial sum of Goldschmidt's *a* and  $\frac{1}{2}c$ . Ungemach's elements are:

$$a:b:c = 0.5396:1:1.0867; \beta = 104^\circ 38'.$$

The structural investigation of lorandite, made by Hofmann (1932), confirmed Ungemach's lattice. Hofmann, however, described it by means of the axial directions of Goldschmidt, whose *c* unit length had to be halved. The plane of symmetry was found to be a glide-plane, with glide-component  $\frac{1}{2}a$ . The space-group symbol was written  $P2/a$ ; its criterion,  $\{h0l\}$  with *h* even. The only forms observed in the  $[010]$  zone are, in Krenner's notation:

$$001.200.401.201.405.$$

TABLE 3. TRANSFORMATION MATRICES FOR LORANDITE SETTINGS

| From \ To                | Krenner                 | Goldschmidt                                                 | Ungemach                                                                            | Hofmann                                                     | Peacock                                                                             |
|--------------------------|-------------------------|-------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------|-------------------------------------------------------------------------------------|
| Krenner<br>1894          | 1 0 0<br>0 1 0<br>0 0 1 | 0 0 1<br>0 $\frac{1}{2}$ 0<br>$\frac{1}{2}$ 0 $\frac{1}{2}$ | $\frac{1}{4}$ 0 $\frac{1}{4}$<br>0 $\frac{1}{2}$ 0<br>$\frac{1}{4}$ 0 $\frac{3}{4}$ | 0 0 1<br>0 $\frac{1}{2}$ 0<br>$\frac{1}{4}$ 0 $\frac{1}{4}$ | $\frac{1}{2}$ 0 $\frac{1}{2}$<br>0 $\frac{1}{2}$ 0<br>$\frac{1}{4}$ 0 $\frac{1}{4}$ |
| Goldschmidt<br>1898      | 1 0 2<br>0 2 0<br>1 0 0 | 1 0 0<br>0 1 0<br>0 0 1                                     | 0 0 $\frac{1}{2}$<br>0 1 0<br>1 0 $\frac{1}{2}$                                     | 1 0 0<br>0 1 0<br>0 0 $\frac{1}{2}$                         | 1 0 1<br>0 1 0<br>0 0 $\frac{1}{2}$                                                 |
| Ungemach<br>1923         | 3 0 1<br>0 2 0<br>1 0 1 | 1 0 1<br>0 1 0<br>2 0 0                                     | 1 0 0<br>0 1 0<br>0 0 1                                                             | 1 0 1<br>0 1 0<br>1 0 0                                     | 1 0 1<br>0 1 0<br>1 0 0                                                             |
| Hofmann<br>1932          | 1 0 4<br>0 2 0<br>1 0 0 | 1 0 0<br>0 1 0<br>0 0 2                                     | 0 0 1<br>0 1 0<br>1 0 1                                                             | 1 0 0<br>0 1 0<br>0 0 1                                     | 1 0 2<br>0 1 0<br>0 0 1                                                             |
| Peacock<br>(unpublished) | 1 0 2<br>0 2 0<br>1 0 2 | 1 0 2<br>0 1 0<br>0 0 2                                     | 0 0 1<br>0 1 0<br>1 0 1                                                             | 1 0 2<br>0 1 0<br>0 0 1                                     | 1 0 0<br>0 1 0<br>0 0 1                                                             |

The choice of axes in the monoclinic system is limited to that of  $a$  and  $c$ , since  $b$  is fixed by the symmetry. The rule is to choose the shortest two lattice translations in the (010) net, the shorter of the two being taken as  $c$ . Now, neither Ungemach's nor Hofmann's cell was chosen according to that convention. Ungemach's unit length  $a$  is indeed the shortest lattice translation in the net; his  $c$ , however, is not the next shorter, but one slightly longer. Hofmann took the shortest translation for his  $c$ , but preferred to retain Goldschmidt's  $a$ , probably on account of it being the glide direction.

Peacock (unpublished) proposes to adhere to the morphologists' convention and adopts the smallest two translations: the shortest one as  $c$  and the next one as  $a$ ; this is the diagonal of Goldschmidt's mesh  $-(\mathbf{a}+\mathbf{c})$ . Peacock's elements are:

$$a:b:c = 1.0873:1:0.5390; \beta = 104^\circ 16'.$$

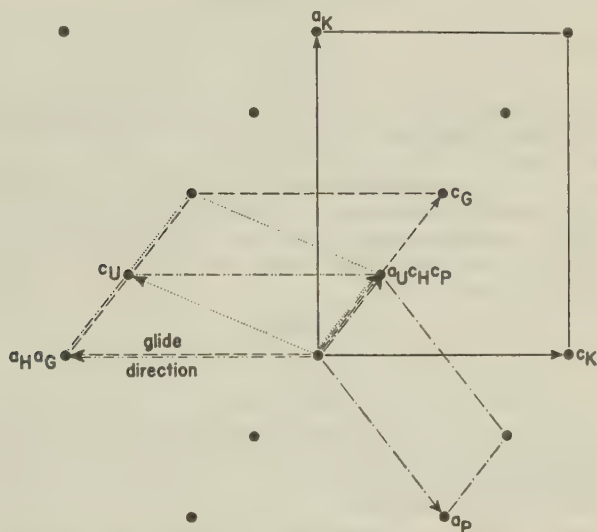


FIG. 4. Direct lattice of lorandite projected on (010). Solid line, Krenner; dashed line, Goldschmidt; dotted line, Ungemach; dash-and-dot, Peacock; dash-and-double-dot, Hofmann. The  $b$  unit-length of Krenner is twice that of the other settings. The negative  $b$  of Ungemach and Peacock is the positive  $b$  of the other settings.

The relationships between the various settings are illustrated in Fig. 4. The transformation matrices are collected in Table 3.

What is the space-group symbol and how will the space-group criterion be expressed in the various settings?

The Krenner and the Goldschmidt settings may be left out since they do not correspond to unit cells of the lattice (the Goldschmidt cell being double; the Krenner cell, octuple).

Hofmann's setting is the simplest. Space-group  $P2/a$ . Glide-component:  $\frac{1}{2}\mathbf{a}$ . Criterion:  $\{h0l\}$  with  $h$  even.

The setting advocated by the *International Tables*, in which the glide direction is made the  $c$  axis, would be equally simple. Space-group:  $P2/c$ . Glide-component:  $\frac{1}{2}\mathbf{c}$ . Criterion:  $\{h0l\}$  with  $l$  even.

The Ungemach setting leads to symbols which are a little more complicated, but which are provided for in the Hermann-Mauguin system of notation. Space-group:  $P2/n$ . Glide-component:  $\frac{1}{2}(c-a)$ . Criterion:  $\{h0l\}$  with  $(l-h)$  even.

In the Peacock setting the glide direction is such that a special Mauguin symbol must be devised. The space-group can be represented by  $P2/x$ , provided the glide-component be explicitly indicated as  $\frac{1}{2}(a+2c)$ . The criterion becomes  $\{h0l\}$  with  $(h+2l)$  even. The application of the convention of the shortest translations leads here to some difficulties: impossibility to take advantage of the Mauguin notation in its simplest form for the space-group symbol, with the resulting necessity of expressing the glide-component; a complicated space-group criterion, which is apt to conceal rather than bring out the systematic "morphological extinctions" of crystal forms. The forms in the zone  $[010]$  are written, in Peacock's notation:

$$201.200.201.001.\bar{6}05,$$

with the criterion " $(h+2l)$  even."

#### *Lanarkite*

In all three preceding examples, the lattice is monoclinic primitive ( $P$ ). The only other monoclinic lattice mode is usually made one-face centered, either  $C$  (base centered) or  $A$  (front pinakoid centered).

Compliance to the rule of the shortest two lattice translations in the  $(010)$  plane may lead to a unit cell that is not referable to any of these modes. Lanarkite is a case in point. The unit cell defined by the shortest translations turns out to be body centered (Richmond and Wolfe, 1938), with space-group  $I2/m$  (no glide-plane). This case is provided for by the Hermann-Mauguin symbols.

## APPENDIX II

### CRYSTAL SYSTEMS OTHER THAN MONOCLINIC

#### *Triclinic system*

The question of space-group symmetry does not enter into consideration. There is never any systematic extinction of  $x$ -ray spectra on the photographic film, nor of crystal forms in the morphological development.

#### *Orthorhombic system*

The structure investigators have devised conventions of their own, in which the relative lengths of the three unit lengths play no part, the setting being governed by the recognition of screw-axes or glide-plane (*International Tables*). Perusal of the recent literature on structural descriptions will show that the structural conventions as to setting are freely disregarded. The rules proposed in this paper entail no inconvenience, since the Hermann-Mauguin space-group notation provides a symbol for any one of the six possible settings. Note that the old convention of orienting antihemihedral crystals with the 2 axis vertical ( $mm2$ ) will be disregarded if the rule  $c < a < b$  is adopted. The symmetry symbol may have to be written  $2mm$  or  $m2m$ , which is not objectionable.

#### *Tetragonal system*

The choice is always limited to two settings, at  $45^\circ$  to each other, the  $c$  axis being imposed by point-group symmetry. One setting corresponds to the smallest cell, either primitive ( $P$ ) or body-centered ( $I$ ); the other, to a multiple cell, either base-centered ( $C$ ) or all-face centered ( $F$ ). The logical course is to adopt the smallest cell in all cases. This procedure will remove the ambiguity that existed in the choice of a setting for the following classes:

$4/m\ 2/m\ 2/m$ ,  $4\ 2\ 2$ ,  $4\ m\ m$ ,  $4/m$ ,  $4$ , and  $\bar{4}$ . In the case of the chalcopyrite class, the morphologists have used the rule that the 2-axes should be taken as coordinate axes. This implies that the symmetry of the class should always be written  $\bar{4}\ 2\ m$ , in which case the unit cell may or may not be the smallest one. The proposal of adopting the smallest cell may then, in some cases, lead to the necessity of writing the symmetry  $\bar{4}\ m\ 2$ , which entails no drawback. In the setting  $\bar{4}\ 2\ m$ , the forms  $\{hhl\}$  are tetragonal disphenoids and the forms  $\{hOl\}$  are tetragonal dipyrramids. In the setting  $\bar{4}\ m\ 2$ , the forms  $\{hhl\}$  become dipyrramids, while the forms  $\{hOl\}$  become disphenoids. The two cases are well recognized in Friedel's *Leçons* (1926).

#### Hexagonal system (*sensu vasto*).

The choice, here as in the tetragonal system, is restricted to two settings. One of them is turned  $30^\circ$  (or  $90^\circ$ ) with respect to the other.

If the lattice is hexagonal (designated by  $C$  in the *International Tables*), the point-group symmetry may be one out of twelve. If the horizontal axes of coordinates  $a_1$ ,  $a_2$ ,  $a_3$ , are taken parallel to the horizontal edges of the smallest hexagonal cell, there will never be any ambiguity. The point-group symmetry of a crystal belonging to the antihemihedry with a  $\bar{6}$ -axis, may be written either  $\bar{6}\ m\ 2$  or  $\bar{6}\ 2\ m$ . The same situation exists for the parahemihedry with a  $\bar{3}$ -axis ( $\bar{3}\ 2/m\ 1$  or  $\bar{3}\ 1\ 2/m$ ), the holoaxial tetartohedry ( $3\ 2\ 1$  or  $3\ 1\ 2$ ), and the antitetartohedry ( $3\ m\ 1$  or  $3\ 1\ m$ ). In each one of these classes, the first setting was the one imposed by former morphological conventions, namely that the 2-axes or the normals to the planes of symmetry be chosen as coordinate axes. This case is similar to that of the class  $\bar{4}2m-\bar{4}m2$ .

If the lattice is rhombohedral (hexagonal- $R$ ), and is referred to the smallest  $R$ -centered hexagonal cell, there are still two alternatives. The dominant rhombohedron may have a face sloping forward, in which case it is symbolized  $\{10\bar{1}1\}$ , or backward, in which case it is indexed  $\{01\bar{1}1\}$ . The second setting may be obtained from the first by a  $180^\circ$  (or  $60^\circ$ ) rotation about the  $c$  axis. In the first setting, the cell (which is here a triple cell) has its additional nodes located at  $\frac{2}{3}\frac{1}{3}\frac{1}{3}$  and  $\frac{1}{3}\frac{2}{3}\frac{2}{3}$ ; in the second setting, at  $\frac{1}{3}\frac{1}{3}\frac{2}{3}$  and  $\frac{2}{3}\frac{2}{3}\frac{1}{3}$ . The extinction criterion of the faces  $(hkl)$  is, in the first setting,  $2h+k+l=3n$  or  $h+i+l=3n$ ; in the second setting,  $2h+k-l=3n$  or  $h+i-l=3n$ . It is unfortunate that the *International Tables* should recommend the adoption of the second setting. The dominant rhombohedron has always been indexed  $\{10\bar{1}1\}$  by mineralogists.

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## GAMAGARITE, A NEW VANADIUM MINERAL FROM THE POSTMASBURG MANGANESE DEPOSITS

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### ABSTRACT

Gamagarite, a new mineral with a composition corresponding to the formula  $\text{Ba}_4(\text{Fe}, \text{Mn})_2\text{V}_4\text{O}_{15}(\text{OH})_2$  has been found in the manganese ores of the Postmasburg district, Cape Province. It has the following properties: Colour, very dark brown. Habit, prismatic.  $H = 4\frac{1}{2} - 5$ . Sp. gr. 4.62. Monoclinic, elongated parallel to  $b$ ,  $b = Y$ .  $\beta = 62^\circ 40'$ .  $X \wedge c = \pm 41^\circ$  in the acute angle  $\beta$ .  $\alpha = 2.016$ ,  $\beta = 2.040$ ,  $\gamma = 2.130$ .  $2V_z = 46^\circ - 62^\circ$ .  $r < v$  fairly strong. Pleochroism, red-brown to light salmon-buff.

### GENERAL

While engaged upon a study of the manganese ores of the Postmasburg district, the writer found a mineral differing in composition and properties from previously described species. This mineral was found only in specimens collected at one locality on the farm Gloucester, situated about sixteen miles north of the village of Postmasburg. It takes its name from the Gamagara ridge, a prominent line of hills running from north to south across the western part of the farm.

The sample of manganese ore in which the gamagarite occurs is of variable texture and in a hand specimen presents a streaked or rudely banded appearance, due to an irregular concentration of the associated minerals. It is composed chiefly of dark iron-gray sitaparite with rose-coloured "books" of ephesite (1), and brown flakes of diasporite (2). In this ore gamagarite is present as aggregates of needles, a centimetre or more in length, and also as somewhat flattened prisms. A more detailed description of the ore specimens follows on a later page.

### PHYSICAL PROPERTIES

The colour is dark brown to nearly black; the luster, adamantine. Its streak is reddish-brown. The mineral is moderately magnetic to the electromagnet.  $H = 4\frac{1}{2} - 5$ . Specific gravity = 4.62. This value is the mean of two determinations made on two portions of the crushed and cleaned mineral. Each portion of about 700 milligrams was weighed in a small pan, first in air and then in water. The values obtained were 4.616 and 4.624. Three cleavages were noted in the prismatic zone; two distinct and a third difficult. Under the microscope, gamagarite is clear but strongly coloured. It is pleochroic in colours varying from light salmon-buff to deep red-brown, and is for the most part crowded with minute opaque inclusions.

## CRYSTALLOGRAPHIC AND OPTICAL PROPERTIES

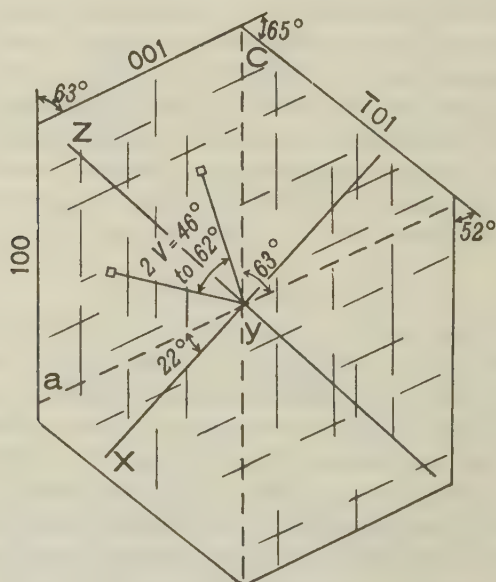
Crystal faces are present on the prisms but these are of poor quality and in part epimorphic. The two cleavage planes are paralleled by crystal faces and gave fair reflections on the goniometer. The third difficult cleavage was observed in one instance on the goniometer and in numerous cases on the universal stage.

Measurements indicate that gamagarite is monoclinic with the prisms elongated parallel to  $b$ ;  $b = Y$ . Extinction angles were impossible to measure with accuracy owing to the imperfect nature of the cleavages. In a number of thin sections, indeed, no cleavage directions could be seen. The two best cleavages were considered as  $\{001\}$  and  $\{100\}$ , and the third as  $\{\bar{1}01\}$ . In the case of flattened crystals it was observed that the largest face is always the base.

The angle between  $\{001\}$  and  $\{100\}$  is  $62^\circ 40' (\pm 30')$  and that between  $\{001\}$  and  $\{\bar{1}01\}$ ,  $65^\circ (\pm 5^\circ)$ . The axial elements, as far as these could be determined are thus as follows:

$$\beta = 62^\circ 40'. \quad c/a = \frac{\sin (001 \wedge \bar{1}01)}{\sin (\bar{1}00 \wedge \bar{1}01)} = 1.15.$$

Figure 1 shows the positions of the various optical and crystallographic directions.



*Optic orientation of gamagarite.*

The extinction angle  $X \wedge c$  in the acute angle  $\beta$  probably varies somewhat, but in view of the indistinctness of the cleavages a study of this variation was not attempted. Out of 11 sections measured, 8 gave values between  $43^\circ$  and  $39^\circ$ . One gave  $37^\circ$  while for the remaining two sections the angle was  $33^\circ$ .

Gamagarite has the following optical properties:

$$\alpha = 2.016, \beta = 2.040, \gamma = 2.130 \text{ (all } \pm .01).$$

$$2V_z = 46^\circ - 62^\circ. r < v, \text{ fairly strong.}$$

Pleochroism,  $X$  = red-brown.

$Y$  = very deep red-brown.

$Z$  = light salmon-buff.

Absorption,  $Y > X > Z$ .

Alpha was measured by immersion of the crushed mineral in liquids of high index prepared according to the method described by West (3). Gamma was measured by embedding in sulphur-selenium melts in the manner previously noted by the writer (4). The following are actual values obtained during the measurement of  $\gamma$ : 2.136, considerably above  $\gamma$ ; 2.132, very near but slightly above; 2.130, very near but slightly above; 2.125, considerably below  $\gamma$ . These figures show that the method yields results of good accuracy within the range in which observations were made. Where necessary, sodium light was used for accurate matching.

$2V$  was measured on the universal stage and the results are considered accurate to about  $4^\circ$ . Variations in indices were not observed. One or more of the indices must, however, vary with  $2V$  and small variations of  $\alpha$  and  $\gamma$  are probable. Since the highest and lowest indices in a crush were measured, the true birefringence may be slightly lower than the value obtained experimentally.

#### CHEMICAL DATA AND DISCUSSION

Only a small proportion of the gamagarite in the rock was pure enough to be suitable for chemical analysis; the mineral for the most part being crowded with inclusions of hematite, cubes of sitaparite, etc. Clean material was obtained from the coarsely crushed rock by selecting the purest fragments. Each fragment was separately powdered and its purity judged by the colour of the powder and by occasional microscopic examination. The amount of impurities in the resultant material was difficult to estimate but probably did not amount to more than 2%. Dr. C. F. J. van der Walt of the Division of Chemical Services kindly undertook the quantitative analysis and his report is given below.

"0.5 gram of the mineral was fused with 6 grams sodium carbonate and the melt taken up with hot water and a little alcohol, to destroy any manganate present. The insoluble mat-

ter was filtered off and washed with a hot, dilute solution of sodium carbonate. The residue contained the barium, iron and manganese, while the filtrate contained the vanadium.

"The filtrate was acidified with sulphuric acid, reduced according to the sulphurous acid method, and the vanadium titrated with decinormal permanganate. The residue was dissolved in hydrochloric acid, carbon dioxide boiled off, and the iron precipitated with ammonia. The precipitate was filtered off, washed with a hot dilute solution of ammonium chloride, dissolved in hydrochloric acid and reprecipitated, etc. The combined filtrates contained the barium and manganese. The ferric hydroxide was dissolved in hydrochloric acid and the iron determined by titration with permanganate after reduction according to the stannous chloride method.

"The barium was precipitated from the filtrate with sulphuric acid and weighed as  $\text{BaSO}_4$ . The manganese was precipitated from the filtrate of the  $\text{BaSO}_4$  according to the phosphate method and weighed as  $\text{Mn}_2\text{P}_2\text{O}_7$ .

"The water was determined according to the Penfield method. The available oxygen was determined by boiling the sample with hydrochloric acid, and the liberated chlorine passed through a solution of potassium iodide. The liberated iodine was titrated with decinormal sodium thiosulphate."

## ANALYTICAL DATA

|                         | 1     | 2    | 3    | 4      |
|-------------------------|-------|------|------|--------|
| BaO                     | 52.4  | .342 | 1.95 | 53.89  |
| FeO                     | 7.6   | .106 | 1.05 | 7.32   |
| MnO                     | 5.5   | .078 |      | 5.24   |
| $\text{Al}_2\text{O}_3$ | nil   |      |      |        |
| $\text{V}_2\text{O}_4$  | 29.1  | .351 | 2.0  | 29.16  |
| $\text{H}_2\text{O}+$   | 1.06  | .118 | .67  | 1.58   |
| O                       | 3.55  | .222 | 1.27 | 2.81   |
|                         | 99.21 |      |      | 100.00 |

1. Analysis by C. F. J. van der Walt.

2. Atomic ratios.

3. Atomic ratios on the basis of  $\text{V}_2\text{O}_4=2$ .

4. Analysis recalculated from the formula  $\text{Ba}_4(\text{Fe}, \text{Mn})_2\text{V}_4\text{O}_{15}(\text{OH})_2$  with  $\text{Fe}/\text{Mn} = .58/.42$ .

Qualitative tests by the writer showed the presence of a trace of calcium, but no lead, copper or arsenic was detected.

In connection with the above table the following observations may be made: (a) a simple ratio exists between the barium and vanadium atoms and the sum of the iron and manganese atoms; (b) the iron and manganese atoms are thus likely to be in a similar state of oxidation and structurally equivalent; (c) since the states of oxidation of the various metal atoms could not be determined, doubt as to the distribution of the available oxygen remains. In the present case it is assumed that vanadium would be in the vanadate condition rather than that vanadyl would exist in the presence of peroxidized manganese.

The available oxygen is sufficient for all the vanadium to be present as vanadate and for about one-half of the sum of the iron and manganese to be in the ferric and manganic states. The weight of the excess oxygen beyond the requirements of the proposed formula is actually small ( $\pm 0.8\%$ ) and may result from a certain amount of weathering which was unnoticed because of the partial opacity and high relief of the mineral.

In order to obtain as accurate a figure as possible for the percentage of available oxygen, Dr. van der Walt carried out determinations on two different samples. In calculating the results (3.46% and 3.65% O), allowance was made for the fact that after boiling with hydrochloric acid the iron was in the ferric state whilst in the analysis it is given as ferrous oxide.

The water in the analysis has been considered as constitutional but there is no proof that it is not either wholly or in part adsorbed. If it were possible that part of the iron and manganese in the mineral is present in the trivalent state then the following alternative formula may be deduced:



A structural investigation with the aid of  $x$ -rays would be of great value in reaching finality with respect to the composition of gamagarite.

The closest relative of gamagarite appears to be the rare and imperfectly known mineral, brackebuschite. In Dana's *System of Mineralogy* the following properties are given for this mineral: composition, perhaps  $\text{R}_3\text{V}_2\text{O}_8 + \text{H}_2\text{O}$ , with R chiefly Pb, also Fe and Mn. Occurs in groups of small prismatic crystals, flattened and vertically striated. Monoclinic? Optically negative? Axial plane perpendicular to striations. Axial angle large. Larsen and Berman (5) state that the mineral is optically positive and give the indices as  $\alpha = 2.28$ ,  $\beta = 2.36$ ,  $\gamma = 2.48$  (all for lithium light).

Precise information on the composition and structure of brackebuschite is still lacking and its relationship to gamagarite cannot therefore be said to be proved.

#### MICROSCOPIC CHARACTERISTICS OF THE ORE SAMPLE

Sitaparite, the chief constituent of the ore, has a crystalline appearance, the individual crystals varying from a few tenths of a millimetre to a few millimetres in diameter. It is idioblastic towards all the other minerals and has a cubic habit. Between crossed nicols it is very weakly anisotropic and shows the typical twinning lamellae described by Schneiderhöhn (6). Lamellar inclusions of diaspore, oriented parallel to the cube faces are sparingly present and minute, rounded grains of hematite are scattered in patches throughout the sitaparite as well as through all the other minerals.

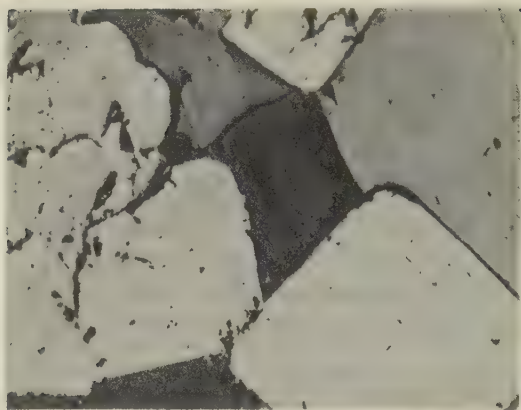


FIG. 2. Polished section showing white sitaparite, gray gamagarite and dark gray ephesite. Mineral in bottom, left-hand corner is diasporite.  $\times 92$ .

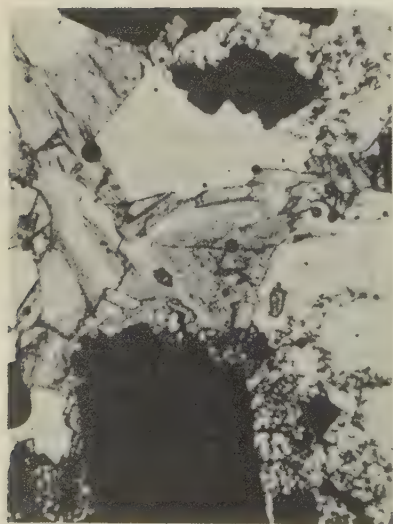


FIG. 3. Thin section showing black sitaparite, gray diasporite and white ephesite.  $\times 34$ .

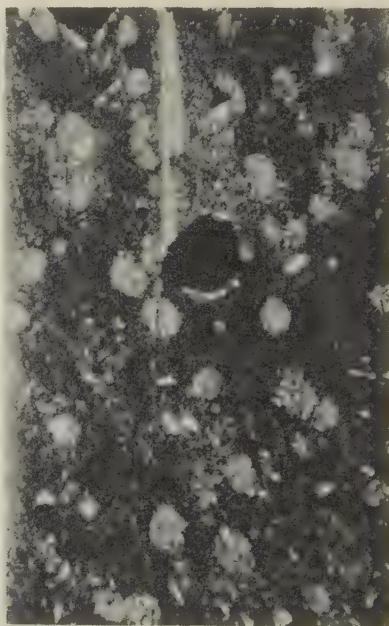


FIG. 4. Thin section of gamagarite with inclusions of hematite showing relict oolitic texture.  $\times 32$ .

Ephesite is present as rose-coloured idiomorphs about 0.5 centimeters in diameter and as fine-grained aggregates, while diasporite forms small, lustrous brown plates. The latter mineral is pleochroic in pink and brownish-green and is usually interstitial to ephesite. Gamagarite is also interstitial to ephesite and may be either idiomorphic or interstitial to diasporite.

Finally, small, hexagonal rods of amesite, a fraction of a millimetre in diameter and a few millimetres in length, are very sparingly present in the rock. It should be mentioned here that a small amount of nickel has been found in a different sample of amesite from the same locality.

The hematite inclusions referred to above, occur in patches and streaks and it is significant that their distribution in part indicates an original oolitic texture. It would thus appear that the ore is a replaced oolitic shale, of which a highly ferruginous variety is common in the vicinity. The conditions under which replacement took place and the source of the replacing solutions are problems intimately connected with the mode of origin of the manganese deposits as a whole. These problems are still being studied.

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## A PRELIMINARY DESCRIPTION OF THE NEW MINERAL PARTRIDGEITE

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Natural  $\text{Mn}_2\text{O}_3$  is found in the Postmasburg manganese ores and has been named partridgeite in memory of the late Mr. F. C. Partridge, formerly Senior Mineralogist of the Geological Survey. It occurs in intimate association with braunite and sitaparite, and also forms isomorphous mixtures with these minerals. Intermediate members of the partridgeite-sitaparite series especially, are well represented.

In a hand specimen partridgeite closely resembles sitaparite. Its colour is dark steel-gray with a yellowish tinge and it has a distinct octahedral or pseudo-octahedral cleavage. Its hardness is about equal to that of braunite or sitaparite. No measurable crystals of partridgeite have yet been found and cleavage fragments have not proved suitable for accurate goniometric measurement.

In a polished section, partridgeite is easily distinguished from braunite by its colour (which is practically identical to that of sitaparite), and from sitaparite by means of etch tests. It may be mentioned here that the composition of South African braunite is given by the formula  $3\text{Mn}_2\text{O}_3 \cdot \text{MnSiO}_3$  and that of South African sitaparite by  $(\text{Mn}, \text{Fe})_2\text{O}_3$ . The percentage of  $\text{Fe}_2\text{O}_3$  in the sitaparite is variable and amounts up to 25.6% have been noted. The colour in polished section of members of the braunite group of minerals apparently varies with the silica content and is little affected by the amount of iron present.

The most useful etch reagent for distinguishing sitaparite from partridgeite is  $\text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$  (equal parts of  $\text{H}_2\text{SO}_4$  (1:1) and  $\text{H}_2\text{O}_2$  (10%)) which strongly etches the latter mineral but has no effect on the former. Etching often reveals an intricate zonal structure in the ferriferous varieties of partridgeite, this zoning probably being due to small chemical differences between the different layers.

Between crossed nicols partridgeite is weakly anisotropic, resembling braunite, while the twinning lamellae characteristic of sitaparite are absent.

Fairly pure specimens of the ferriferous variety of partridgeite have been available for analysis, but the iron-free mineral has thus far been found only in association with braunite and pyrolusite. Three samples have been analyzed by Dr. C. F. J. van der Walt of the Division of Chemical Services. Of these, No. 1 is a ferriferous partridgeite while No. 2 is a braunite-partridgeite-pyrolusite ore. Number 3 was submitted for analysis at a later stage than the previous two samples and was found

to contain a trace of hematite in addition to pyrolusite and braunite. In each of the analyzed specimens the amount of the impurities was estimated by inspection of three polished surfaces under the reflecting microscope.

TABLE 1. ESTIMATED COMPOSITION OF THE SPECIMENS

| No. | Partridgeite | Braunite | Pyrolusite | Diaspore |
|-----|--------------|----------|------------|----------|
| 1   | 92           | 2        | 4          | 2        |
| 2   | 53           | 40       | 7          | —        |
| 3   | 80           | 5        | 15         | —        |

TABLE 2. ANALYSES OF PARTRIDGEITE

| No.                            | 1                       | 2     | 3    |
|--------------------------------|-------------------------|-------|------|
| MnO <sub>2</sub>               | 53.02                   | 56.50 | 61.9 |
| MnO                            | 37.90                   | 35.96 | 35.9 |
| Fe <sub>2</sub> O <sub>3</sub> | 6.71                    | 0.56  | 0.5  |
| SiO <sub>2</sub>               | 0.36                    | 4.92  | 0.5  |
| Al <sub>2</sub> O <sub>3</sub> | 1.49                    | 0.26  | 0.4  |
| H <sub>2</sub> O+              | —                       | 1.0   | —    |
| B <sub>2</sub> O <sub>3</sub>  | —                       | 0.6   | —    |
| CaO                            | —                       | trace | nil  |
| Total                          | 99.48<br>Sp. Gr. = 4.96 | 99.80 | 99.2 |

From the above analyses the approximate mineralogical constitution of the specimens has been calculated, assuming the formula of partridgeite to be (Mn, Fe)<sub>2</sub>O<sub>3</sub>. The small amounts of Al<sub>2</sub>O<sub>3</sub> (in No. 2), H<sub>2</sub>O, B<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> (in No. 3) have been neglected in these calculations.

TABLE 3. CALCULATED COMPOSITION OF THE SPECIMENS

|   | Partridgeite | Braunite | Pyrolusite | Diaspore |
|---|--------------|----------|------------|----------|
| 1 | 87           | 4        | 7          | 2        |
| 2 | 29           | 50       | 19         | —        |
| 3 | 75           | 5        | 19         | —        |

The agreement between the observed and the calculated values for the composition of samples 1 and 3 is satisfactory but in case of sample No. 2 there is a rather large discrepancy. This is no doubt in large part due to the low accuracy of the method of estimating the composition of

the specimens, but it is also possible that part of the silica given in the analysis is contained in the partridgeite molecule. The analysis of this sample is of interest chiefly because it shows that a mineral superficially resembling sitavite is practically free from iron.

Boron was detected in sample No. 2 with the aid of the spectrograph by Dr. B. Wasserstein of the Union Geological Survey. As shown by subsequent tests this element is contained chiefly in the braunite. The quantitative determination of  $B_2O_3$  in various samples of braunite ore was undertaken by van der Walt and results as high as 1.1% were obtained.

## NOTES AND NEWS

### A UNIQUE OCCURRENCE OF BOBIERRITE, $\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$

JOHN W. GRUNER AND C. R. STAUFFER, *University of Minnesota, Minneapolis, Minnesota.*

Bobierite has been found in several places as small crystals in guano, and also as nodules. The occurrence described here is in a fossil elephant tusk. The tusk belongs to the species *Archidiskodon imperator* (Leidy) the type of which was found in Pleistocene gravels of Nebraska. The specimen under discussion was found in 1941 in a gravel pit of the Hallett Construction Company one mile southwest of Edgerton, Pipestone County, Minnesota. It was on top and partly embedded in a hard bouldery blue clay (probably Nebraskan till) which is overlain by 9 to 11 feet of terrace gravels and 3 feet of fine black soil, the whole being a terrace along Rock River. The tusk, the front end of which is missing, is about 7 feet long and 8 inches in diameter at the larger end. It tapers about half an inch in its whole length. When found, the middle part was embedded in the clay, and the curved ends projected into the gravel. The portion in the clay was unusually well preserved, but the ends in the gravel were badly decomposed. As drying occurred, the outer shell,  $\frac{3}{8}$  inch thick, peeled off the tusk. Between this outer shell and the inner core, the layers of bobierite were found.

There is really only one important layer of the mineral about 1 to 2 mm. thick, but thinner ones separated by paper-thin ivory partitions may be seen. The main layer is made up of three parts, a middle portion consisting of large well crystallized bobierite and two border zones which consist of minute fibrous aggregates. The fibers point toward the center and are white in color. These zones are not more than two to three-tenths of a millimeter thick and may become almost invisible. The center layer consists of rosette-like flattened aggregates. The cleavage (010) of the crystals is commonly more nearly normal to the layer than parallel to it. The largest cleavage pieces may be several millimeters long and half a millimeter wide. They are practically colorless.

The physical properties of the mineral are: perfect cleavage parallel (010), hardness 2, specific gravity 2.2 (with Jolly balance). Optically +, axial plane parallel to (010),  $Y=b$ ,  $Z \wedge c = 28^\circ$ . Indices very close to those given by Larson and Berman,<sup>1</sup>  $\alpha = 1.510$ ,  $\beta = 1.520$ ,  $\gamma = 1.543$ . The x-ray powder photograph is very similar to that of vivianite.

The mineral dissolves rapidly in cold HCl. No chemical analysis of the mineral has been made, but tests for Fe, Al, and Ca show merely traces

<sup>1</sup> U. S. Geol. Survey Bull. 848, p. 101 (1934).

of these elements. The absence of calcium was carefully checked by Dr. R. B. Ellestad, to whom the writers are indebted for his help. It is significant that a magnesium phosphate can develop from calcium phosphate—the ivory has the structure of apatite—to the complete exclusion of calcium. This fact is particularly interesting as the mineral *hautefeuilite* described by M. L. Michel<sup>2</sup> is said to be isomorphous with *bobierite*. It has been given the formula  $(\text{Mg}, \text{Ca})_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ . Its actual analysis showed 5.71% CaO according to Michel.

The absence of Fe in the *bobierite* needs some explanation since it is isomorphous with *vivianite*  $\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ .<sup>3</sup> Either the ground water which brought in the Mg contained no Fe or the latter was in the ferric state, which is improbable.

<sup>2</sup> *Bull. soc. franc. mineral.*, **16**, 40 (1893).

<sup>3</sup> Barth has also called attention to this in this journal, **22**, 338 (1937).

## BOOK REVIEW

ECONOMIC MINERAL DEPOSITS by ALAN M. BATEMAN, Professor of Economic Geology at Yale University. 898 pages, John Wiley & Sons, Inc., 1942. Price \$6.50.

This textbook deals with the origin and occurrences of mineral resources. The discussion of General Principles and Processes (Part I) constitutes the first half of the book and the remainder is almost evenly divided between Metallic Mineral Deposits (Part II), and Non-metallic Mineral Deposits (Part III).

The first four chapters of Part I cover general discussions of mineral deposits and include (1) Introduction, (2) Brief History of Economic Geology, (3) Materials and Modes of Formation, and (4) Relation to Magmas. These chapters comprise a résumé of the economic aspects of mineralogy, petrology, and general geology.

Chapter 5 contains the principal contributions of this book as a text in economic geology for the detailed discussion of Processes of Formation of Mineral Deposits covers 237 pages. The treatment follows a classification proposed by the author which is outlined under the following headings: magmatic concentration, sublimation, contact metamorphism, metasomatic replacement, cavity filling, sedimentation (exclusive of evaporation), evaporation, mechanical concentration, residual concentration, oxidation and supergene enrichment, and metamorphism. While most texts in economic geology contain much of the same material concerning the formation of mineral deposits, this chapter is undoubtedly one of the better presentations of the concepts because of its clearness, scope, and organization.

The next two chapters in Part I contain discussions of Controls of Mineral Localization, and Folding and Faulting of Mineral Deposits. The remainder of Part I consists of synopses on Classification of Mineral Deposits; International Relations and Conservation in Minerals; Geology in Prospecting, Exploration, Development, and Valuation of Mineral Deposits; and on Extraction of Metals and Minerals. The inclusion of these chapters in Part I makes it sufficiently comprehensive and independent that it might serve as an introduction to the subject of economic geology. This opinion differs somewhat from that of the author for he suggests that "For short courses in economic geology Parts II and III can be used separately from Part I. For longer courses all three parts can be used."

Part II considers the occurrences of various metallic mineral deposits and follows the customary arrangement of Precious Metals, Nonferrous Metals, Iron and Ferroalloy Metals, and Minor Metals. Most of the discussions are general and include (1) history, (2) production and distribution, (3) mineralogy, tenor, treatment, and uses, (4) kinds of deposits and origin, (5) examples of deposits in the United States, and (6) important deposits in other countries.

Part III concerns non-metallic mineral deposits and consists of brief discussions which include: (1) properties and uses, (2) production and distribution, (3) occurrence and origin, (4) extraction and preparation, and (5) examples of deposits. This part is subdivided into ten chapters on the basis of the principal uses in order to lend emphasis to the recent utilizations of these resources. Ceramic Materials, Metallurgical and Refractory Material, Industrial and Manufacturing Materials, and Ground Water Supplies are chapter headings which indicate the character of some of the trends. The other chapters are those which one would expect, such as Structural and Building Materials, Fertilizers, Abrasives, etc. Most of the chapters in Part III are 15-20 pages in length, but the one on Mineral Fuels contains 20 pages on coal and 40 pages on petroleum.

Selected references follow the discussion of each mineral resource and although the lists are not lengthy, they appear to be adequate for general purposes.

There are about 300 illustrations which include diagrams, cross-sections, and a few maps. Most of them have been well chosen and a number are new to the textbook field.

The text of the book proper contains 860 pages and the preface states that "Its chief purpose is as an elementary textbook, but it could be adapted almost equally well to more advanced courses. . . . The use of this book presupposes some knowledge of general geology and mineralogy." Difficulties may arise, however, in using it for elementary work, unless the students have had good preparation in mineralogy and petrology.

*Economic Mineral Deposits* adds to the variety of textbooks on economic geology by placing a new emphasis on principles and processes rather than by innovations concerning the geologic description of various districts and deposits.

CARL E. DUTTON

## PROCEEDINGS OF SOCIETIES

### NEW YORK MINERALOGICAL CLUB, INC.

#### *Minutes of meeting held on Oct. 21, 1942*

Professor William Lawrence Bragg, Dr. Leonard James Spencer, and Mr. Herbert P. Whitlock were elected to honorary membership by a unanimous vote of the members attending. Brief biographies of each of these men were read by Dr. Pough.

After the reading of annual committee reports, the balance of the meeting was devoted to talks by the members on their summer collecting experiences. The speakers were: Messrs. Morgan, Sampter, Northup, McKeown, Maynard, Marcin, and Trainer. One of the more outstanding specimens exhibited was Mr. Trainer's pseudomorph of quartz and epidote after large garnet crystals.

#### *Meeting of Nov. 18, 1942*

Miss Elizabeth Armstrong of the Department of Geology of Columbia University addressed the meeting on "Crystal Quartz in the Eastern United States." The crystallography of quartz was reviewed with special reference to the distinction between right- and left-handed crystals and the various types of twins. She then described the manner of occurrence of quartz crystals at Ellenville, N. Y., Herkimer and Montgomery Counties,

N. Y., Allegheny, Alexander, and Iredell Counties, N. C. and the Hot Springs, Arkansas, district. The descriptions were illustrated with colored slides taken during the course of her study of these localities.

*Meeting of Dec. 18, 1942*

The meeting was addressed by Dr. A. F. Buddington whose subject was "The Rate of Progress in Mineralogy and Its Significance." Based on the rate of progress in mineralogy during the last 40 years, Dr. Buddington predicted that at least one new field of mineralogic science may be expected to develop on the average every 10 years. The average length of time for a major new technique or theory to have its body of data developed and incorporated into a textbook, adequate for routine use, is from 20 to 25 years. There has been as much progress in the mineralogic sciences in the past 50 years as in all previous time, and there is no indication of any decrease in the present rate of expansion. Perhaps at least half of our progress in the United States has been through the importation of new ideas and techniques, often with considerable delay due to language barriers. The proposal was made that some organization should select the best foreign papers each year, translate them into English, and republish them.

*Meeting of Jan. 27, 1943*

Dr. Pough announced for the Education Committee that a course of 6 classes in mineral identification would be given on Saturday afternoons at the museum.

Letters of thanks for their having been elected to honorary membership were read from Prof. W. L. Bragg, Dr. L. J. Spencer, and Mr. H. P. Whitlock.

Dr. Alonzo Quinn of Brown University then spoke on "The Origin of Rhode Island Minerals." There are three major rock types in the state, the oldest being a set of metamorphics, then a younger set of igneous intrusives, mostly granites, and a still younger series of sedimentaries. Dr. Quinn discussed various localities with respect to the types of rock in which the minerals are found and its influence on paragenesis. The localities described included: South Foster, the Manton Ave. quarry in Providence, the Harris and Dexter quarries, Copper Mine Hill, Iron Mine Hill, and Cranston. Numerous specimens were exhibited, among them being: bowenite from the Harris quarry, flattened quartz crystals from the Dexter quarry, and some notable greenish sphene crystals from the Manton Ave. quarry.

*Meeting of Feb. 17, 1943*

Dr. Harry Berman of Harvard University spoke on the "New Classification of Minerals." After discussing the general aspects of setting up any classification and the criteria used to test its adequacy, Dr. Berman went on to describe the classification adopted for the new edition of *A System of Mineralogy*.

The principal units of classification, called classes, are chemical and were introduced mainly for mineralogical reasons. They include sulfides, sulfo salts, simple oxides, uranium oxides, etc. These classes are then divided into types based on the ratio of total positive elements to negative elements or groups, with the higher ratios first. Types are further divided into groups, as, the galena groups, and the sphalerite group of the type AX in the class of sulfides. These groups are further divided into series and species.

An attempt has been made to reduce the number of mineral names by dropping separate names for varieties, except such terms as ruby, sapphire, etc. of exceedingly long standing. Wherever possible descriptive terms are preferred for varieties instead of separate names. Thus, "ferroan spinel" instead of "ceylonite" etc.

Dr. Berman stated that arrangements have been completed for the publication of the first volume within the next few months.

M. ALLEN NORTHUP, *Secretary*.